

**INVESTIGATION ON THE PROCESSING OF WHEAT BRAN AND CONDENSED  
DISTILLERS SOLUBLES AS ANIMAL FEED**

A Thesis

Submitted to the College of Graduate Studies and Research

in partial fulfillment of the requirements

for the degree of

**Master of Science**

in the

Department of Chemical and Biological Engineering

University of Saskatchewan

Saskatoon, Saskatchewan

by

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## **ABSTRACT**

Owing to the increasing demand for alternative fuel additives, the Canadian ethanol industry has grown tremendously. In Western Canada, wheat has been the dominant feedstock for ethanol production because of steadily increasing domestic production and higher ethanol yields. Low protein and high starch wheat varieties have further increased the potential of grain-based ethanol production. As a consequence, the increased ethanol production has demonstrated an exponential increase in the availability of its co-products. Depending on the processes used, several co-products are produced, such as bran, condensed distillers solubles (CDS), and distillers dried grains with solubles (DDGS). Wheat bran is obtained as the co-product when debranning is incorporated in ethanol production.

Debranning of wheat feedstock may be integrated into the ethanol production process to improve ethanol quality and yield. Debranning follows the principles of abrasion and friction. It improves the starch content of the feedstock and the fermentation efficiency of the ethanol plants. Several abrasive equipment that generate products having good quality and desirable ethanol yield are being used commercially. Among these, the Satake mill and the tangential abrasive dehulling device (TADD) are prominent, having high debranning efficiency, levels of sanitation, and improved production rates. In this thesis, the laboratory debranning process using these two equipment was optimized by varying the process variables in order to improve the ethanol production process. In the Satake mill, the sample size (30 and 200 g), rotational speed (1215, 1412, and 1515 rpm), grit size (30, 36, and 40), and retention time (30, 60, and 90 s) were varied. In the TADD mill, the sample size (30 and 200 g), grit size (30, 36, 50, and 80), and retention time (120, 180, 240, and 300 s) were varied while maintaining a constant rotational

speed of 900 rpm. The experimental results indicated that in the Satake mill, 200 g sample size, 1515 rpm rotational speed, 30 grit size, and 60 s retention time provided optimal debranning and starch separation efficiency. For the TADD mill, 200 g sample size, 900 rpm rotational speed, 50 grit size, and 240 s retention time provided optimal results.

Increased availability of ethanol co-products from the pretreatments and other processes brings forth the need for broadening the areas of application of these co-products. Among the various applications, the usage of the co-products as animal feed is predominant. Ethanol co-products have been traditionally incorporated as ingredients for animal feed. This thesis is aimed at combining the wheat bran and CDS in varying proportions (70:30, 80:20, and 90:10) and producing high quality animal feed pellets. Laboratory-scale pelleting was done at varying pelleting temperatures, 60, 75, and 90°C, to optimize the pelleting process. The results of laboratory-scale single pelleting indicated that 90:10 bran-CDS ratio and 90°C pelleting temperature produced pellets having good physical properties. Pilot-scale pelleting was done to verify the optimized variables, and to produce dimensionally stable and highly durable feed pellets. The results showed that 70:30 bran-CDS mixture produced pellets with high nutrient content and physical properties ( $760.88 \pm 2.04 \text{ kg/m}^3$  bulk density and  $97.79 \pm 0.76\%$  durability). Similar to the single pelleting results, high pelleting temperatures (75°C) produced pellets with desirable physical properties. However, on cooling, the bulk density and durability change was the highest for 70:30 bran-CDS pellets, indicating an improvement in the physical characteristics. In conclusion, the bran and CDS, the two co-products of the ethanol industry, could be combined to produce feed pellets having good physical and nutritional properties.

## **ACKNOWLEDGEMENT**

First and foremost, I would like to thank Almighty God for his grace, blessings, and eternal guidance.

I would like to express my heartfelt gratitude to Dr. Lope G. Tabil for his invaluable guidance and constant encouragement at each and every step of my thesis work. He exposed me to the research topic through rigorous discussions and constantly provided me with timely support and suitable suggestions.

I am also grateful to my co-supervisor, Dr. Venkatesh Meda for his technical guidance and valuable contributions throughout the research program. I sincerely thank each member of my graduate advisory committee, Dr. Oon-Doo Baik and Dr. Charles Maule, for their supervision and motivation throughout my research. Many thanks to the Feed Opportunities in the Bioethanol Industries (FOBI) network under the Agricultural Bioproducts Innovation Program (ABIP) of Agriculture and Agri-Food Canada for funding my project work. I acknowledge the College of Graduate Studies and Research for the travel award and financial support throughout my studies.

The technical assistance of Louis Roth of the Department of Chemical and Biological Engineering, and Ravindra Heendeniya and Dr. Colleen Christensen of FOBI are acknowledged. Material used in this project was kindly provided by Terra Grain Fuels of Belle Plaine, SK.

I would like to thank my Parents, siblings, Jiji, Jinu, Manoj, and Lijin, and my nieces for trusting in me and keeping me in their prayers constantly. I would like to thank my friends in Saskatoon for the faith that they had in the successful completion of my research. Last, but not the least, I thank my former University, Karunya University, for providing me an opportunity to interact with the U of S professors and to pursue my graduate program under their supervision.

## DEDICATION

*I would like to dedicate this thesis to my loving family who encouraged me with their love  
and kept me constantly in their prayers.*

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## LIST OF ABBREVIATIONS

AACC	American Association of Cereal Chemists
ANOVA	Analysis of Variance
ASAE	American Society of Agricultural Engineers
Ca	Calcium
CDS	Condensed distillers solubles
CO <sub>2</sub>	Carbon dioxide
CP	Crude protein
CPS	Canadian Prairie Spring
CRFA	Canadian Renewable Fuels Association
DDG	Dried distillers grains
DDGS	Distillers dried grains with solubles
DDS	Distillers dried solubles
DIP	Degradable intake protein
DM	Dry matter
FAO	Food and Agriculture Organization
FAOSTAT	FAO Statistical database
GLM	General linear model
HRSW	Hard red spring wheat
IFIF	International Feed Industry Federation
K	Potassium
MC	Moisture content

Mg	Magnesium
NDF	Neutral detergent fiber
NDSU	North Dakota State University
NEg	Net energy for growth
NEm	Net energy for maintenance
NIR	Near Infrared reflectance
P	Phosphorus
RH	Relative humidity
rpm	Revolutions per minute
S	Sulfur
SK	Saskatchewan
TADD	Tangential abrasive dehulling device
TDN	Total dietary nutrients
UIP	Undegradable intake protein
UN	United Nations
wb	Wet basis
WDG	Wet distiller grains
WDGS	Wet distillers grains with solubles



# **CHAPTER ONE**

## **INTRODUCTION AND OBJECTIVES**

### **1.1 Introduction**

Ethanol is a renewable fuel, derived by processing feedstock such as corn, wheat, and sugarcane. It is used as engine fuel and fuel additive, and is gaining recognition rapidly. The commercialization of ethanol is affected by factors such as increasing energy prices and the environment-based negative effects of fossil fuels. As a result of the benefits associated with ethanol usage, the ethanol industry has shown significant expansion in the past few decades. In 1998, the global ethanol production was 31.2 billion L (Berg 1999), and in 2009, it was 73.9 billion L (RFA 2011a). On reviewing the trend in the production, it was estimated that, by 2010, the production of ethanol would reach 85.9 billion L, a 16.2% increase from 2009 (Mousdale 2011).

Canada realized the true potential of ethanol a little later when compared to the other international markets. In 2004, the ethanol production capacity was only 0.24 billion L/year in Canada, 92% of which was grain-based (Arachchilage 2011). For improving the production capacity, the federal Ethanol Expansion Program (EEP) funded the development of more ethanol plants [(S and T)<sup>2</sup> Consultants Inc. 2004]. It aimed at 1 billion L/year production capacity (Olar et al. 2004). The venture was successful and by the year 2011, Canada reached an ethanol capacity of 1.8 billion L (Hooper 2011). Constant efforts are being taken to increase the production further. The main focus is on grain-based ethanol production which contributes significantly to Canada's economy and makes use of two main processes for ethanol production.

In general, the ethanol production is done using the wet milling and dry milling of grains. In the former, the grain kernel is separated into its parts (bran, germ, and endosperm) prior to the fermentation step. In the latter, the whole grain is milled and used for fermentation. Ethanol is produced from the starch component, whereas, the fiber and protein are removed in the form of co-products. Feedstock having low protein and high starch content are preferred for ethanol production (Sosulski and Sosulski 1994). Grains, such as corn, wheat, sorghum, and barley have high starch content, and are used as ethanol feedstock in different regions of the world.

In Canada, a higher production, availability, and yield capacity makes wheat the first choice of ethanol producers (Racz 2008). The majority of it is grown in the provinces of Western Canada, such as Saskatchewan, Manitoba, and Alberta. In Canada, only seven ethanol plants were in operation in 2004. Further developments lead to improvement in the commercial production of ethanol. With the use of improved technologies, the production costs of ethanol could be reduced, benefiting the population. Statistics showed that the production was 231 million L in 2005 (Olfert and Weseen 2007) and the capacity was 1.8 billion L in 2011 (Hooper 2011) for Canada. Similarly, the Western Canadian ethanol production also increased in the past years. As of September 2007, the production capacity was 180 million L (Racz 2008). Table 1.1 shows the status of ethanol development in Canada in 2010, as reported by the Canadian Renewable Fuels Association (2010). For improvement in the productivity, the processes favoring production economics are selected. Due to this reason, the dry milling process using wheat as feedstock is dominant in the Canadian ethanol industry.

Table 1.1 Ethanol: Canadian Production List (Canadian Renewables Fuels Association 2010)

Plant	City	Province	Feedstock	Capacity (MMLy)**	Status
Alberta Ethanol and Biodiesel GP Ltd.	Innisfail	AB	Wheat	150	Proposed Plant
Amaizeingly Green Products L.P.	Collingwood	ON	Corn	58	Operational
Atlantec Bioenergy	Milford	NS	Energy Beets	n/a	Demo Facility
Enerkem Alberta Biofuels-Edmonton Waste-to-Biofuels Facility	Edmonton	AB	Municipal Solid Waste (landfill waste)	36	Under Construction
Enerkem Inc. – Sherbrooke Pilot Plant	Sherbrooke	QC	Various Feedstocks	0.5	Demo Facility
Enerkem Inc. -Westbury Commercial- Demo. Facility	Westbury	QC	Wood Waste	5	Demo Facility
GreenField Ethanol Inc. Chatham	Chatham	ON	Corn	195*	Demo Facility
GreenField Ethanol Inc. Johnstown	Johnstown	ON	Corn	230	Operational
GreenField Ethanol Inc. Tiverton	Tiverton	ON	Corn	27*	Operational
GreenField Ethanol Inc. Varennes	Varennes	QC	Corn	155	Operational
Growing Power Hairy Hill	Hairy Hill	AB	Wheat	40	Proposed Plant
Husky Energy Inc. Lloydminster	Lloydminster	SK	Wheat	130	Operational
Husky Energy Inc. Minnedosa	Minnedosa	MB	Wheat and Corn	130	Operational
IGPC Ethanol Inc.	Aylmer	ON	Corn	162	Operational
Iogen Corporation	Ottawa	ON	Wheat and Barley Straw	2	Demo Facility
Kawartha Ethanol Inc.	Havelock	ON	Corn	80	Operational
NorAmera BioEnergy Corp.	Weyburn	SK	Wheat	25	Operational
North West Terminal Ltd.	Unity	SK	Wheat	25	Operational
Permolux International, L.P.	Red Deer	AB	Wheat, Wheat Starch, Corn, Barley, Rye, Triticale	42	Operational
Pound-Maker Agventures Ltd.	Lanigan	SK	Wheat	12	Operational
Suncor St. Clair Ethanol Plant	Sarnia	ON	Corn	400	Operational
Terra Grain Fuels Inc.	Belle Plaine	SK	Wheat	150	Operational

\* Volumes include industrial alcohol production, \*\*MMLy- million liters per year

The ethanol production process for wheat and corn is much the same. Although the yield capacity of corn is higher, the quantity of the co-products produced in wheat-based ethanol production compensate for its lower yield capacity. Wheat has a yield of 370 L/t [(S and T)<sup>2</sup> Consultants Inc. 2003] and 38% of distillers dried grains of the feedstock, whereas, corn has a yield of 400 L/t [(S and T)<sup>2</sup> Consultants Inc. 2003] and 32% of distillers dried grains. The yield of ethanol from wheat and corn is 1287 and 1514.2 L/acre (Veal and Chinn 2007). This implies that grain-based ethanol production is also useful in generating co-products having good market value (Mathews and McConnell 2009). On a mass basis, the ethanol, CO<sub>2</sub>, and the co-products produced are almost equivalent. For each tonne of grain, almost 365 L of ethanol, 290 kg of CO<sub>2</sub>, and 290 kg of dried distillers grains with solubles (DDGS) are produced (Racz 2008). Grain-based ethanol production also provides an alternative market to wheat growers, improving the local economy. The utilization of wheat classes lower in protein content for ethanol production increased the utilization of these wheat varieties. However, the expansion in the ethanol production also brings forth some issues in the commercial sector which is discussed below.

The expansion in ethanol production leads to excessive availability of the co-products, such as distillers dried grains (DDG), condensed distillers solubles (CDS), and DDGS. There is a need of practical applications of these co-products. As a result, feed manufacturers started using the ethanol co-products as feed alternatives. The following reasons also support this action. The commercially available feed mixtures comprise of almost 40-50% grains (Dhakad et al. 2002), using a large portion of the grains produced worldwide. Also, the competition of feed grains for ethanol production has increased the need for feed alternatives. Since feed demand is influenced by factors like feed prices and livestock population (Rosegrant et al. 2001), using grains as feed ingredient has increased animal feed costs. With rapidly growing world population, the demand

for animal-based food further increased feed prices (FAO/IFIF 2010) because of increased feed demand. This also led feed processors and the animal industry to increasingly explore alternate feed materials. Incorporating ethanol co-products as feed ingredients was an obvious way to increase profit and improve the revenue in feed manufacturing. According to the ANAC (2009), the Canadian feed industry sales reached \$4 billion. Studies focusing on the utility of the co-products as livestock feed ingredients have shown high energy values of DDGS, equivalent to corn, in finishing cattle (Trenkle 2004). Similar avenues for the other co-products, such as CDS and DDG are also being explored.

The nutritional profile of CDS is very appealing, having high mineral, vitamin, and protein content (20-30% dry matter basis; Lardy 2007). To avoid the usage of dry ration, CDS is added as the liquid component to reduce fines. NCRRP (1984) also stated that CDS is the major protein source for ruminants, and it improves their digestibility. It has been used in low-quality feeds, and is extensively used for swine (Squire et al. 2005), when they were fed wet feeds. It has a moisture content higher than DDGS, and provides fatty acids to poultry, proteins to cattle (NCRRP 1984), and vitamins and minerals to swine (Lardy and Anderson 2009). The typical composition of corn CDS (fresh and stored) was reported by Braun and de Lange (2004), from pig farms using liquid feeding systems (Table 1.2). The CDS were stored in storage tanks on the farm for varying periods of time, ranging from 1 day up to several weeks.

Wheat bran is another co-product which is being used as animal feed ingredient. It is consumed as a source of digestible fiber for dairy livestock, sheep, swine, and other ruminants. The relative feeding value of wheat is approximately 91-106% of corn, 109-114% of barley, and equivalent to sorghum (Maner 1985). Survey reports state that desirable animal performance and weight gain are not attained solely by forage consumption. There is need for other energy

supplements, such as cereal grains. But cereal grains affect rumen fermentability by degrading the proteins in the feeds (Mustafa et al. 2000a).

Table 1.2 Nutritional composition of fresh and stored corn condensed distillers soluble (CDS) samples collected on commercial pig farms in Ontario, Canada (100% dry matter basis, Braun and de Lange 2004)

<b>Nutrient</b>	<b>Fresh CDS</b>	<b>Stored CDS</b>
No. of samples	5	5
Dry matter, %	30.5± 0.58 (29.7-31.1)	27.2± 3.58 (22.5-31.2)
Crude protein, %	22.3± 1.28 (20.8-24.1)	25.2± 1.63 (23.5-27.8)
Crude fat, %	18.9± 1.36 (17.4-20.9)	22.4± 1.23 (20.7-23.7)
Ash, %	8.4± 0.59 (7.8-9.1)	10.0± 1.09 (9.0-11.8)
Ca, %	0.04± 0.01 (0.02-0.06)	0.06± 0.01 (0.04-0.07)
P, %	1.43± 0.12 (1.25-1.58)	1.64± 0.15 (1.47-1.85)
Na, %	0.21± 0.04 (0.15-0.27)	0.21± 0.03 (0.18-0.25)
pH	3.7± 0.2 (3.5-3.9)	3.5± 0.1 (3.4-3.6)
Acetic acid, %	0.11± 0.02 (0.08-0.13)	1.66± 1.67 (0.32-4.53)
Propionic acid, %	0.63± 0.10 (0.50-0.76)	0.88± 0.27 (0.69-1.33)
Butyric acid, %	0.01± 0 (0.01-0.01)	0.01± 0.01 (0.01-0.01)
Lactic acid, % <sup>1</sup>	9.8	15.4
Total non-starch polysaccharides, %	6.1± 0.2 (5.9-6.3)	5.5± 1.2 (3.5-6.7)
Starch, %	9.9± 2.0 (7.7-12.2)	6.8± 1.1 (5.1-7.9)
Total sugars, %	3.5± 0.3 (3.2-4.0)	1.2± 1.2 (0-2.7)

<sup>1</sup>Lactic acid was determined from pooled samples; the numbers after ± represent the standard deviation values and the numbers in parenthesis represent the range

Unlike cereal grains, wheat bran increases energy intake, without these negative effects (Horn and McCollum 1987, as cited in Hess et al. 1996), and improves animal performance. The

wheat bran phytase activity ranges from 2349 to 9945 PU/kg (Eeckhout and Depaepe 1994; Viveros et al. 2000; Steiner et al. 2007). A high phytase activity triggers an increase in phosphorus consumption in swine (Darwazeh 2010). Due to these reasons, wheat bran is being recognized as an ingredient in feed formulation (Paik 2003).

For the production of the wheat bran, debranning is used. It is a pre-milling treatment, used for controlled removal of the wheat grain's bran layers. It ensures that grain kernels remain intact (Mousia et al. 2004), which is associated with the nutrient quantity and quality of the grains (Dexter and Wood 1996). It removes up to 17% of the grains, corresponding to the entire bran content in the grain kernel. Studies have shown its effect on thermal properties (Mousia et al. 2004) and bread baking performance (Lin et al. 2010). Debranning succeeded due to improvement in abrasive material efficiency and reduced energy consumption. Newly developed debranning equipment have vertical configuration, unlike older versions (Dexter and Marchylo 2001). Two of the equipment, the Satake mill and the tangential abrasive dehulling device (TADD), are being commercially explored due to their high production rates.

The utilization of the above mentioned co-products such as, wheat bran, CDS, and others, can affect the rumen pH negatively. To prevent this, improved methods of processing these co-products are necessary. Pelleting is a processing technique that has numerous benefits. It is an energy-intensive process (Fairfield 2003), affecting feed mill economics. It is defined as the method of producing pellets from premixed feed ingredients. Pelleting affects the physical quality of the pellets (Thomas et al. 1997). Although it is expensive due to high capital and maintenance costs, its advantages make it a preferable choice to improve the animal performance and feed efficiency.

To summarize, with the increased availability of co-products from ethanol production, they may be used as alternative feeds to fulfill the nutrient requirement for healthy growth of animals. Feeds enhancing the animal performance are essential. Wheat bran and CDS, co-products of ethanol production, have high nutritive value, and are considered in this study as alternative feed. Pelleting is the process adapted in this study to add value to co-products of ethanol production. Thus, the goal of this study is to find processing opportunities of the co-products of ethanol production which use wheat as the feedstock.

## **1.2 Objectives of Research**

This research project aims to improve debranning and the pelleting methods and conditions to ensure optimal production, energy efficiency, and cost competitiveness in an ethanol plant that uses wheat as feedstock. The specific objectives are:

1. to optimize the debranning process using wheat in the two milling equipment, the Satake mill and the tangential abrasive dehulling device (TADD), before the wheat is used as feedstock for ethanol production.
2. to determine the effect of combining bran and condensed distillers solubles (CDS) on the physical and nutritional properties of the pellets produced; and
3. to optimize the pelleting process by producing durable and dimensionally stable feed pellets.

It is envisioned that the investigation will contribute towards the development of an alternate feed having high nutritional quality, better flowability, and improved physico-chemical characteristics. Another contribution of this study will be determining the conditions leading to



improvement in the debranning efficiency of the abrasive mills and optimal pelleting in the pilot-scale pellet mill.

The research project was implemented in two phases, with the first phase addressing the first objective and the second phase addressing the other two objectives.

**Phase I** consisted of debranning of wheat in the Satake mill and the TADD for optimizing the debranning conditions and comparing the debranning efficiency of the two mills.

**Phase II** consisted of preparation of bran-CDS samples in varying ratios and pelleting the sample mixtures to produce dimensionally stable and durable animal feed pellets.

### **1.3 Organization of the Thesis**

The thesis is organized according to the University of Saskatchewan guidelines for manuscript-based theses. Chapter 1 in this thesis introduces the subject matter and the objectives of the research. The review of literature of ethanol and wheat production, debranning process and equipment, animal feed processing, and pelleting are discussed in Chapter 2. Chapter 3 presents the study on optimizing the debranning of wheat, including the method, materials and equipment used, results, and discussion. Chapter 4 presents the study on the pelleting characteristics of bran and CDS, including method, materials and equipment used, results, and discussion. Chapter 5 provides a general discussion of all the results obtained in the previous chapters. Chapter 6 concludes the thesis by summarizing the main observations based on the results discussed in preceding chapters. Some important recommendations for future studies and commercial development of the product are given in Chapter 7.

## **CHAPTER TWO**

### **REVIEW OF LITERATURE**

The literature on wheat production, ethanol production, debranning of wheat, utilization of condensed distillers solubles (CDS), animal feed production, and animal feed pelleting techniques required for understanding, prior to developing animal feed, are reviewed and presented in this chapter.

#### **2.1 Introduction**

Worldwide, the benefits associated with utilization of cereal grains as feed ingredient have been recognized. Many animal feeds that can help in animal growth remain unused, or have low usage commercially (Makkar 2002). Additionally, insufficiency of quality feeds negatively affect animal productivity (Faizi et al. 2004). The main reason for this is inadequate knowledge of the nutritional advantages associated with their usage (Makkar 2002). Owing to the food versus fuel debate and increasing world population, feed producers have shown considerable interest in finding alternatives to cereal grains as a feed ingredient for livestock and poultry feeds lately (Gilbert 2004). Table 2.1 shows the nutrient profile of feedstuff being used currently. Ethanol co-products have been used as livestock feed ingredient for many years. But lately, they have been recognized as an economical alternative to high priced grain-based feeds (Mathews and McConnell 2009). Trenkle (1996) stated that using ethanol co-products in cattle feeding would increase the economic growth.

Table 2.1 Nutrient profiles of selected feedstuffs (Mathews and McConnell 2009)

	<b>Dry matter</b>	<b>Crude protein</b>	<b>Total digestible nutrients</b>	<b>Lysine</b>	<b>Methionine</b>	<b>Sulfur</b>
	<b>Percent</b>					
Corn	87	7.5	80	0.24	0.18	0.08
Soybean meal	88	47.8	79	0.71	0.70	0.43
Dried distillers grain with solubles	92	27	82	0.80	0.51	0.30
Wet distillers grains with solubles	34.9	31	91			
Corn gluten feed	88	21	75	0.60	0.50	0.16

Among the co-products, CDS is a well-known source of fat, minerals, and proteins for ruminants (NCRRP 1984). It is being used in corn gluten feed (Pesta 2011), and as a liquid supplement. Lardy (2007) analyzed the CDS composition and found that on a dry matter basis, CDS has 20-30% protein, 85-90% total digestible nutrients, and high sulfur content. Popularly known as “syrup”, CDS is also used as an energy source, and acts as a binder to condition dry feeds (Lardy 2007). Through experiments conducted in South Dakota State University, it was concluded that diets comprising of 10% CDS improved weight gain (Trenkle 2004). Similar to CDS, wheat bran, a co-product of wheat milling industry, has also been used as an animal feed ingredient. Bran is the source of dietary fibers and proteins, and is used for cattle and swine feeds. It is obtained by the debranning of wheat.

Debranning removes the bran layers progressively (Tkac 1992) without damaging the grain kernels. It uses friction, abrasion, or a combination of both to separate the outer layers from the endosperm (Lijuan et al. 2007). Wheat, the preferred feedstock for ethanol production in

Canada, is debranned using different debranning equipment to obtain the bran fractions. Debranning reduces the required conditioning time for seed coat removal from 12-36 h to 20 min (Andersson 2011). Newer methods also incorporate controlled pressures (Singh and Singh 2010) during debranning for shortening the debranning time, thereby increasing the production rates. On obtaining the different feed ingredients, they should be processed to make them applicable as animal feeds.

The animal feed ingredients are processed using different techniques (FAO/IFIF 2010), making them suitable for animal consumption. For instance, processing the animal feed ingredients into pellets enhances the feed quality (Thomas et al. 1998) overcoming the problems associated with conventional feeds. Usage of heat processing during pelleting reduces pathogens (Winowiski 1995) and improves starch digestibility (Svihus et al. 2005), enhancing the animal performance. Although the literature presents various topics related to animal feed production using ethanol co-products, this thesis will primarily focus on combining two such animal feed ingredients, namely wheat bran and CDS, forming animal feed pellets having good physico-chemical properties.

## **2.2 Ethanol Production**

The present global scenario of ethanol production provides a better understanding of the availability of the co-products as animal feeds.

Ethanol, a form of biomass energy, is an essentiality in today's world. Biomass energy provides benefits like renewable fuel supply, lower greenhouse gas emissions (Hahn-Hagerdal et al. 2006), economic growth, and new venues of biomass usage (Lior 2008). Biomass has the potential of developing new arenas in biological engineering (Rabson and Rogers 1981). In

Canada, hydroelectric power is the primary source of renewable energy and biomass energy is the secondary source (NRC 2009). Biomass is directly converted into liquid fuels, i.e. biofuels, for transportation needs. Biofuels reduce 12% greenhouse gas emissions (Hill et al. 2006), as compared to the fossil fuels. Ethanol and biodiesel are the two biofuels being used commercially. Out of the two, ethanol is currently produced from lower-value grades of grain such as corn or wheat, or sugar source such as sugarcane (Mani et al. 2002). Table 2.2 shows the grain production scenario for Canada in the last eight years.

It is clear that the production of wheat grains is the highest in the Canadian prairies. Thus, it is important to utilize wheat in different applications, such as human food, animal feed, and as ethanol feedstock. Based on the production statistics, it can also be seen that corn is not used as the predominant ethanol feedstock in Canada, owing to a very low productivity. With increased demand for ethanol in Canada, different wheat varieties are being grown for ethanol production purpose. Among them, the soft white wheat, with lower protein and higher starch content, is suitable for ethanol production (Sosulski and Sosulski 1994).

### **2.2.1 Global scenario**

Due to public policies, ethanol as a fuel additive and as a fuel has become popular. Many countries around the world are taking steps to furnish the energy needs by using renewable fuels and increasing the local fuel production. In 1998, 31.2 billion L of ethanol was produced worldwide (Berg 1999). It reached 17 billion L in 2000 and more than 52 billion L in 2007. In the world ranking for fuel ethanol production from 2007 to 2009, the U.S ranked first and Canada ranked sixth (Table 2.3).

Table 2.2: Grain production statistics in Canada (Canada Grains Council Database 2007; Estimates are based on last 8 years of production)

<b>Production ('000 t)</b>					
		<b>Manitoba</b>	<b>Alberta</b>	<b>Saskatchewan</b>	<b>Canada</b>
<b>Hard red spring</b>	2006- 2007	3543	6966	9076	18617
<b>Wheat</b>	07/08(P)	2659	5019	5935	14098
	Est./Year	3200	5500	8900	19000
<b>Oats</b>	06/07	979	670	1526	3602
	07/08(P)	1230	763	2587	5086
	Est./Year	950	850	1550	3600
<b>Barley</b>	06/07	1145	4626	3570	9573
	07/08(P)	1336	5356	4406	11847
	Est./Year	1250	5400	4950	12800
<b>Durum</b>	06/07	8.7	691	3946	4162
	Est./Year	12	850	3800	4700
<b>Winter Wheat</b>	06/07	571.5	160	275	3301
	07/08(P)	781	231	508	2694
	Est./Year	-	-	-	
<b>Triticale+(Rye)</b>	04/05	7.6	(86.4)	28(103)	45(165)
<b>Corn</b>	03/04	495	17	-	

\*P stands for Projected

### 2.2.2 Production process

Ethanol usage has benefits such as no greenhouse gas emissions reduction in fossil fuel utilization, and net energy return (Shapouri et al. 2002). In order to improve grain-based ethanol yield, ethanol producers mainly use the wet milling and dry milling process (Singh and Eckhoff 1997). In the dry milling process, the whole grain is milled and processed for ethanol production

(Sosulski and Sosulski 1994). In contrast, the first step in wet milling is conditioning the grains by “steeping” (RFA 2011b). Steeping is the process of conditioning the grains (Kreider and Curtiss 2007), to separate the grain into its component. It is done by soaking the grains in water and dilute sulfurous acid (RFA 2011b) for 24 to 48 h. Upon separation of the germ, the remaining grain kernel is centrifuged to separate the starch and fiber components. The starch is processed for ethanol production whereas the fiber and protein components are removed in the form of co-products. The co-products from wet milling have high market value and are a main-stream commodity.

Table 2.3: Annual fuel ethanol production for 2007-2009 (Millions of US liquid gallons per year)

<b>World rank</b>	<b>Country</b>	<b>2007</b>	<b>2008</b>	<b>2009</b>
1	U.S	6,498.60	9,000.00	10,750.00
2	Brazil	5,019.20	6,472.20	6,577.89
3	European Union	570.30	733.60	1,039.52
4	China	486.00	501.90	541.55
5	Thailand	79.20	89.80	435.20
6	Canada	211.30	237.70	290.59
7	India	52.80	66.00	91.67
8	Columbia	74.90	79.30	83.21
9	Australia	26.40	26.40	56.80
10	Others	247.27		
<b>Total</b>		<b>13,101.70</b>	<b>17,335.29</b>	<b>19,534.99</b>

Table adapted from: “2007 and 2008 World fuel ethanol production”. F.O. Licht, as cited in RFA (2005) and “2009 Global ethanol production (Million Gallons)”. F.O. Licht, cited in RFA (2010).

In the U.S., wet milling is dominant, while Canada uses dry milling (Agriculture and Agri-Food Canada 2002). Corn is the first choice for ethanol production in North America,

having high yield (Mani et al. 2002). In Canada, wheat is used (Warren et al. 1994), due to its large production, especially in Western Canada. Table 2.4 is a comparison of feedstock supply and ethanol demand in different provinces in Canada. The feedstock supply in the Canadian prairies is much higher than the demand for ethanol. Due to these reasons, almost 66% of the ethanol production comes from the prairie wheat (Baron 2008).

Table 2.4: Comparison of feedstock supply and ethanol demand [(S and T)<sup>2</sup> Consultants Inc. 2003]

	<b>Ethanol Demand (million L)</b>	<b>Corn (million L)</b>	<b>Wheat (million L)</b>	<b>Barley (million L)</b>	<b>Potatoes (million L)</b>	<b>Total Feedstock Supply (million L)</b>
<b>B.C.</b>	162	0	0	0	0	0
<b>Alberta</b>	181	0	400	74	0	474
<b>Sask.</b>	61	0	666	60	0	726
<b>Manitoba</b>	51	0	266	15	0	281
<b>Ontario</b>	539	400	0	0	0	400
<b>Quebec</b>	300	120	0	0	0	120
<b>N.B.</b>	110	0	0	0	3.5	3.5
<b>N.S.</b>	37	0	0	0	0	0
<b>P.E.I.</b>	8	0	0	15	6	21
<b>Nfdl.</b>	21	0	0	0	0	0
<b>Yukon</b>	2	0	0	0	0	0
<b>NWT</b>	1	0	0	0	0	0
<b>Nunavut</b>	0	0	0	0	0	0
<b>Canada</b>	1,473	520	1332	164	9.5	2,025.5



Ethanol production process using grains as the feedstock is distributed into five steps: 1) processing of feedstock; 2) saccharification; 3) fermentation; 4) distillation; and 5) recovery and processing of co-products (Thiessen 2001). The first step in ethanol production is grain milling. As mentioned earlier, in wet milling, steeping is done prior to grain milling. The grains are screened to remove stones, weeds, and other debris. The screened grains are ground into course flour, which is sent for saccharification. Saccharification converts the starch in the ground grains into simple sugars. Enzymes act as the process catalyst in saccharification. Fermentation follows, where yeasts like *Saccharomyces cerevisiae* catalyze the breakdown of the sugars to ethanol. CO<sub>2</sub> is the waste given off (Singh et al. 2001). Fermentation is done at specific conditions of temperature and pH. Product removal, simultaneous to fermentation, increases the ethanol yield (Haraldson and Rosen 1982) and production rate. It also reduces accumulation of the inhibitory products (Yang and Tsao 1995). The next step is distillation. It works on the principle of difference in the boiling point of two liquids. In distillation, evaporation at 78°C separates the ethanol from the ethanol solution (Widyaratne 2005; Rendleman and Shapouri 2007). It separates the liquids with significantly different or similar boiling points (fractional distillation, Qadri et al. 2009). Fractional distillation achieves up to 95-96% ethanol. Distillation is energy-intensive and it removes the water-soluble components (Rausch and Belyea 2006). The water-soluble components known as whole stillage (McKinnon et al. 2008) contain the fiber, protein, fat, and minerals (Bothast and Schlicher 2005). The whole stillage is 10 to 15 times the ethanol produced (volume basis, Maiorella et al. 1983, as cited in Widyaratne 2005). Since ethanol is made up of the starch component of the feedstock, it comprises of one-third portion of the distillation products. The portion of the feedstock forming the whole stillage is two-third of the

distillation product, and is produced in large amounts. Its dry matter content (5-7%) is affected by feedstock used and the fermentation efficiency (Thiessen 2001).

### **2.2.3 Ethanol production co-products**

The ethanol production process emphasizes on distillation, the process separating ethanol and whole stillage. It is an important part of the production process, as it ensures extraction of almost 90-95% ethanol by volume (Taherzadeh and Karimi 2007). The whole stillage is centrifuged to attain wet distiller grains (WDG) and thin (clarified) stillage (Mustafa et al. 2000a). Multiple evaporation of thin stillage produces condensed distillers solubles (CDS). WDG from corn has almost 30% dry matter and on drying, it forms dried distillers grains (DDG) (Lardy 2007). The co-products, both in their wet and dried state, are used as animal feed (Larson et al. 1993). WDG contains 50-75% carbohydrate (Mustafa et al. 2000a) and the thin stillage contains 4-8% dry matter, based on the grain used as feedstock (Mustafa et al. 1998). The average nutrient composition of the corn-based ethanol co-products, a compilation of various studies, is given in Table 2.5. The crude protein (CP), total dietary nutrients (TDN), energy required for maintenance (NEm), and net energy required for growth (NEg) levels in DDG, DDGS, and CDS were almost equivalent. High CP levels increase the application of ethanol co-products as feed ingredients. The DM% in CDS and DDG varied substantially. A low UIP in CDS indicated that CDS was not a preferred source of bypass protein. However, a higher moisture content of CDS increases its utilization as the liquid supplement in feed diets. Therefore, both thin stillage and CDS are useful as animal feed ingredient.

Table 2.5: Nutrient composition of corn-based ethanol co-products (Lardy 2007)

<b>Nutrient</b>	<b>Dried Distillers Grains</b>	<b>Dried Distillers Grains plus Solubles</b>	<b>Modified Wet Distillers Grains plus Solubles</b>	<b>Wet Distillers Grains plus Solubles</b>	<b>Condensed Distillers Solubles</b>
DM, %	88 to 90	88 to 90	50	25 to 35	23 to 45
<b>DM Basis</b>					
TDN, %	77 to 88	85 to 90	70 to 110	70 to 110	75 to 120
NEm, Mcal/cwt	89 to 100	98 to 100	90 to 110	90 to 110	100 to 115
NEg, Mcal/cwt	67 to 70	68 to 70	70 to 80	70 to 80	80 to 93
CP, %	25 to 35	25 to 32	30 to 35	30 to 35	20 to 30
DIP, % CP	40 to 50	43 to 53	45 to 53	45 to 53	80.0
UIP, % CP	50 to 60	47 to 57	47 to 57	47 to 57	20.0
Fat, %	8 to 12	8 to 12	12 to 15	10 to 18	9 to 15
Calcium, %	0.11 to 0.20	0.10 to 0.20	0.02 to 0.03	0.02 to 0.03	0.03 to 0.17
Phosphorus, %	0.40 to 1.15	0.40 to 0.80	0.50 to 1.42	0.50 to 0.80	1.30 to 1.45
Potassium, %	0.49 to 1.08	0.87 to 1.33	0.70 to 1.00	0.50 to 1.00	1.75 to 2.25

DM= dry matter, TDN= total dietary nutrients, NEm= Net energy required for maintenance, NEg= Net energy required for growth, CP= crude protein, DIP= degradable intake protein, UIP= undegradable intake protein.

Table adapted from: Stock, et al. (1995); Tjardes and Wright (2002); NRC (2001); Iowa Renewable Fuels Association. [www.iowafa.org/ethanol\\_coproducts.php](http://www.iowafa.org/ethanol_coproducts.php). Accessed June 19, 2007, Internal laboratory analysis at NDSU. The analyses given in this publication are a range of published values and regionally available laboratory analyses. Products vary and this may not represent what a particular plant is producing at any given time.

Wheat thin stillage is abundant in crude proteins (Mustafa et al. 2000a), whereas, corn thin stillage has very low protein values. In general, studies reported that grain-based thin stillage has low starch content. Contradicting results were reported by Larson et al. (1993) and Ham et

al. (1994) for corn thin stillage. According to the values reported by them, corn thin stillage had abnormally high starch content. Mustafa et al. (2000a) reported that amino acid content in thin stillage and the feedstock used were equivalent. These factors cause the reutilization of thin stillage. Almost 15% of it is used as process water (Kwiatkowski et al. 2006). The remaining thin stillage is evaporated to produce CDS (Ganesan et al. 2006) which can be dried to obtain distillers dried solubles (DDS).

DDG and DDS are marketed as feed rations individually, or in combination, as wet distillers grains with solubles (WDGS), which upon drying is termed as dried distillers grains with soluble (DDGS). DDGS has a good nutrient profile, making it an excellent feed supplement as it acts as a source of proteins and energy for dairy and beef cattle. Similar to the thin stillage composition, wheat DDG have high neutral detergent fiber (NDF) (Mustafa et al. 2000a) and crude protein, and low starch values. The same study also showed that corn DDG had lower NDF, higher starch, and equivalent crude protein values. The availability of neutral detergent insoluble proteins affects the NDF content in grains (Mustafa et al. 1999). DDGS has a high phosphorus content and acts as the supplemental inorganic phosphorus source

### **2.3 Cereal Grains**

In North America, ethanol production is highly dependent on cereal grain supply. Cereal grains are preferred as feedstock in North America because of their high starch content (Sosulski and Sosulski 1994). Cereal grains have also been the major energy, caloric, protein, and nutrient source for humans and animals (Cordain 1999). Grain-based ethanol co-products have high nutritive value and studies on their effect on the dairy and beef cattle industry have been done (Lardy 2007). Animal feeds based on grain-based ethanol co-products have been highly

recommended. Some of the ethanol co-products are corn (Lodge et al. 1997ab; Al-Suwaiegh et al. 2002), wheat (Iwanchysko et al. 1999; Fisher et al. 1999), and barley (Mustafa et al. 1999 and 2000ab) based.

Based on the many applications of cereal grains, in Canada, grain-based ethanol production represents 92% of the total production capacity (Olar et al. 2004). Wheat is the predominant cereal grain in Canada, because it has the highest production number in terms of area cultivated and the weight harvested.

### 2.3.1 Wheat global production

Wheat (*Triticum aestivum* L.), one of the most important cereal in the world, is the staple food in many countries. According to UN-FAO (2010), global production of wheat was approximately 680 million t in 2010. World wheat production from the main production areas between 2005 and 2010 is given in Table 2.6.

Table 2.6: Global wheat production statistics (in million metric t, FAOSTAT. Available at [faostat.fao.org](http://faostat.fao.org))

Country	2005	2006	2007	2008	2009	2010
EU	135.4	126.7	120.1	150.3	138.7	136.5
China	96.3	104.5	109.9	112.5	115.0	115.2
India	72.0	69.4	74.9	78.6	80.7	80.7
Russia	47.6	45.0	49.4	63.7	61.7	41.5
United States	57.1	57.3	53.6	68.0	60.3	60.1
Canada	25.6	27.3	20.6	28.6	26.5	23.2
<b>World Total</b>	628.7	605.9	607.0	683.4	685.6	651.4

Canada, ranked sixth, had a production of 23.2 million metric t of wheat in 2010. The total Canadian wheat production was forecast to reach 24.08 million t (2011) from 23.2 million t in (2010) (Statistics Canada 2011), a 4% increase. This increase could be influenced by the demand of wheat for human, animals, and as ethanol feedstock. The different varieties of wheat produced are suitable for different purposes, fulfilling all its demands. It also helps in increasing the areas of utility of the wheat grains, helping wheat growers.

#### **2.3.1.1 Composition of wheat grain**

The wheat grain kernel comprises of three main parts: the bran (13-17%), germ (2-3%), and endosperm (80-85%) (Belderok et al. 2000, as cited in Sramkova et al. 2009). Typically, the wheat kernel composition is 12% water, 70% carbohydrates, 12% protein, 2% fat, 1.8% minerals, and 2.2% crude fibers (Pomeranz 1988). In general, the bran is rich in fiber, vitamin B, and ash (mineral) content. On a dry matter basis, proteins and carbohydrates constitute 16% and 80% of the bran (Nandini and Salimath 2001). The germ has 25% protein, 4-5% minerals, and 8-13% lipids (Sramkova et al. 2009). The endosperm comprises of 82% carbohydrates, 1.5% fats, 13% protein, 0.5% ash, and 1.5% dietary fiber (Sramkova et al. 2009). With a high nutritional content, the consumption of wheat and its co-products provides many health benefits for humans and animals.

#### **2.3.1.2 Milling of wheat**

In the early days, the milling process involved crushing the grains for flour production, damaging all parts of the grain kernel. This gave way to newer milling techniques, aimed at separating the endosperm from the bran and germ layers (Jackson and Shandera 1995). It was

appropriately termed as “debranning”. Debranning can help in improving flour characteristics. Chen and Siebenmorgen (1996) used a commercial-scale Satake mill (model BA-7) and emphasised that the degree of milling influenced quality factors like microbial contamination and starch gelatinization. Dexter and Wood (1996) described debranning as “the process of removal of the outer hull layers, allowing the recovery of intact kernels”. Moreover, Bottega et al. (2009) stated that debranning reduced the amount of broken kernels. A study examined wheat fractions and concluded that debranning improved the phenolic and antioxidant quantity in wheat (Beta et al. 2005). Unfortunately, the presence of the crease on the ventral side of the wheat kernel prevents complete debranning of the grain and affects the flour yield (Posner and Hibbs 1997).

## **2.4 Debranning Operation**

Debranning is based on the principles of friction and abrasion (Lijuan et al. 2007), and can be carried out using the combination of the two. During friction debranning, the grains rub against each other, causing the removal of the outer layers (Satake 1969). Grain kernels are rubbed on a rough stone (Hemery et al. 2007) in abrasive debranning. Taylor et al. (1939, as cited in Lawton and Faubion 1989) suggested that the dehulling efficiency could be an indicator of kernel hardness. On abrasion, the grain starch contents increased by 12.2%, as determined by Wang et al. (1997).

### **2.4.1 Advantages of debranning**

Technological developments in debranning operations are currently needed (Scudamore 2008). The methods of milling of wheat to produce flours have been investigated for many years. It involves heat and moisture generation in the mill, piling up flour residues on moisture

condensation (Berghofer et al. 2003). Microorganisms thrive on these residues, contaminating the milling products. Pandiella et al. (2004) used the Satake mill to show that debranning prevented this to a large extent. The experiment showed that, as a result of the debranning (only 4%), almost 87% microbes were eliminated.

Debranning also causes progressive removal of the bran (Tkac 1992), with almost complete recovery of bran layers (Liyana-Pathirana et al. 2006). Debranning has been used successfully in improving flour quality and baking performance. Numerous studies emphasizing the benefits have been conducted. Along with lessened fiber and tannin content (Deshpande et al. 1982a), debranning improved the physical quality, functional properties (Deshpande et al. 1982b), and digestibility of grains. Finer flour particles (Dexter et al. 1994; Sekon et al. 1992; and Singh et al. 1998) and uniform particle size distribution (Mousia et al. 2004) was observed when the effect of bran on flour quality was studied. This, in turn, influenced the bread-making process (Pandiella et al. 2004). Debranning was used to remove the bran layers, keeping the aleurone layers attached to the endosperm. At the same time, debranning was also selected to isolate aleurone-rich fractions (Brouns et al. 2012) to produce nutritionally fortified cereal-based foods.

#### **2.4.2 Debranning equipment**

Many debranning equipment, operating on the principle of abrasion, are being used commercially. Unlike the conventional equipment aimed at milling alone, these equipment allow sequential bran separation such that the bran can be used for other purposes (Tkac 1995). Abrasive mills used for milling sorghum had a higher starch recovery (61-70%) (Higiro et al. 2003). On the other hand, the  $\alpha$ -amylase activity of sprouted wheat reduced upon abrasion



(Chakraborty et al. 2003). Reduction in the negative effects associated with pre-mature sprouting (Singh 2008), and improvement in flour quality and dough strength (Sekhon et al. 1992) were also observed upon debranning. Debranning, prior to the fermentation step of ethanol production, holds many benefits and is advantageous for ethanol production. Ponnampalam et al. (2004) observed an increased fermentation capacity, higher yield, and market value of co-products resulting from debranning. Figure 2.1 show the basic operations in ethanol production including the dehulling process.

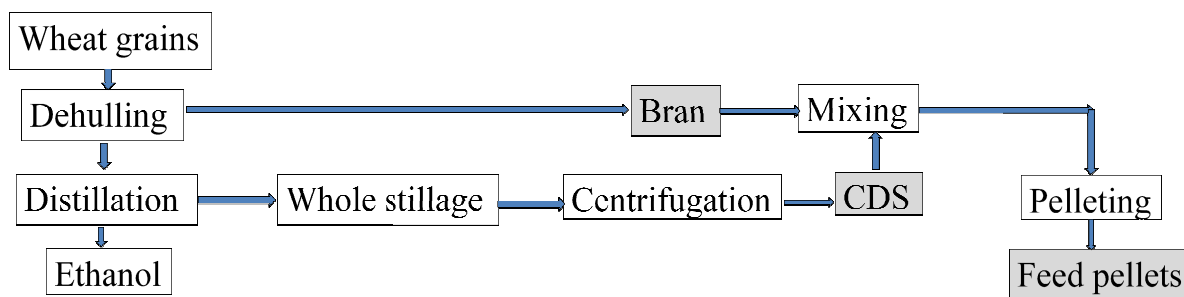


Figure 2.1. Unit operations in ethanol production with the inclusion of dehulling

Debranning optimizes starch digestion and improves the process by removing fermentation inhibitors. Furthermore, by removing the bran layers, the endosperm is more accessible by the enzymes used in the fermentation process (Corredor et al. 2006). Debranning also changes the chemical composition of the distillers dried grains with solubles (DDGS) in corn grains (Ponnampalam et al. 2004). Some commercially used abrasive dehullers are the barley pearler (Rooney and Sullins 1969; Wiesenborn et al. 2003), the Satake grain testing mill (Wang et al 1997; Yeung and Vasanthan 2001), the modified Udy grinding mill (Shepherd 1979), and the tangential abrasive dehulling device (TADD). Among these, the Satake mill and TADD have been investigated, and their advantages have been noted.

#### **2.4.2.1 Satake mill**

The mill has an abrasive roller, made up of carborandum or emery (Liu 2007), installed on a shaft within the milling chamber of the machine. The chamber has a metallic casting and a smooth peripheral surface (Opoku et al. 2003). The materials enter the chamber through a feed hopper and undergo two operations. Initially, the grains fall into the abrasion chamber, between the roller and the metallic casting (George et al. 2010), and get abraded. Passing the abrasion section, the grains move into the friction section. As the grains reach the discharge end, friction between the kernels and the screen lead to debranning. As the process ends, the bran and the debranned kernels get collected into individual chambers (Opoku et al. 2003). Grit size of the abrasive roller, the revolutions per minute (rpm), and retention time are some of the factors affecting the debranning efficiency (Tkac 1995).

The Satake mill is preferred for removing bran and germ from cereal grains. Debranning of grains using the Satake mill improves milling (Meinardus 1985). Reichert et al. (1986) stated that the Satake mill is one of the most commonly used laboratory dehullers. Fang and Campbell (2002 and 2003), and Campbell and Webb (2001) conducted experiments using the Satake STR-100 test roller mill to determine the milling parameters affecting oat breakage. Their results indicated that the separation ratio and chemical composition of the bran were optimal on dehulling, in accordance with the American Association of Cereal Chemists (AACC) standards. The rotational movement of materials in the Satake mill causes frequent contact of the grains and the metallic casting, improving the debranning efficiency. Moreover, a wide area for abrasive contact is advantageous (Satake 1969). According to Dexter and Wood (1996), conditioning the grains for 3-5 min before milling, makes the grain moisture content suitable for milling. Yang

and Seib (1996) found similar results for sorghum grains i.e. conditioning prepares the grains for milling. With higher yield and lower contamination, the purification process following milling may get eliminated. These, in turn, improve the process economics. Lab-scale studies of red gram and soybean also used the Satake mill (Swamy et al. 1991; Sachan et al. 1993) to measure seed coat yield. These advantages make the Satake mill suitable for wheat debranning process.

#### **2.4.2.2 Tangential abrasive dehulling device (TADD)**

The tangential abrasive dehulling device (TADD) works on the principle of tangential abrasion (Reichert et al. 1986), and has been used by Oomah et al. (1981) to dehull barley kernels. TADD is a compact machine having low maintenance (Oomah et al. 1981). A horizontally placed abrasive roller, a stationary aluminum head plate, and eight vertically placed stainless steel open-bottomed sample cups are the main parts (Opoku et al. 2003). The bottomless sample cups rest on the surface of the disc. The cover plate is placed over the cups during the milling operation. Upon coming in contact with the roller, the grains are debranned (Reichert et al. 1986). The device collects and cleans the debranned grain kernels, removing any residual fine material present (Mwasaru et al. 1988). Variations can be brought in the speed of rotation, the grit size of the abrasive roller, clearance between the bottom of the sample cups and the roller, and the residence time. The clearance between the bottom of the sample cups and the roller affects the bran yield (Reichert et al. 1986) and the amount of grains which escape debranning. After milling for a specified duration, the debranned grains are collected using a vacuum aspirator (Opoku et al. 2003). The husk and bran are collected using a cyclone separator (George et al. 2010).

The TADD mill allows controlled removal of the bran layers (Reichert et al. 1986), with less damage to the kernels (Reichert and Youngs 1976). Normand et al. (1965) used the TADD on different cereal grains to study their protein structure. It has also been used in works designed to increase the dehulling quality and seed coat durability (Anglani 1998). Oomah and Mazza (1997) used the TADD to study the physico-chemical properties of flax seeds. They reported high oil and protein values, and low carbohydrate values in the debranned flaxseed. Similar to the Satake mill, TADD was also used for studies on seed coat yield of field peas (Singh et al. 1992). Wehling et al. (1993) established the correlation between the near infrared reflectance (NIR) measurements and the TADD values for yellow dent corn to predict the dry milling characteristics. The TADD has been examined thoroughly, and is used for commercial purposes globally.

## **2.5 Animal Feed**

Many of the grain-based co-products are being used extensively as livestock feed ingredients. They act as the source of proteins (Mathews and McConnell 2009), minerals, dietary fiber (Ge et al. 2000), and vitamins. With the increased demand for high quality animal feed, feed manufacturers are developing better alternatives to the currently available animal feeds. Table 2.7 provides the statistics on global feed production and usage from 1975 to 2001. From 1975 to 2001, there had been constant increase in feed utilization whereas feed production slightly decreased in 1998 and onwards. This could be due to several factors such as technical difficulties, insufficient grain production for feed processing, and variation in the economic conditions (Gilbert 2004).

Table 2.7: Global feed population, feed manufacture and per capita use (Gilbert 2004)

<b>Year</b> <b>(million tons)</b>	<b>Population feed use</b>	<b>Manufactured feed</b>	<b>Per capita</b> <b>(billions)</b> <b>(kg/person)</b>
1975	4.1	290	71
1980	4.5	370	82
1985	4.9	440	90
1990	5.3	537	101
1995	5.6	590	105
1996	5.7	597	105
1997	5.8	605	104
1998	5.9	575	97
1999	6.0	586	98
2000	6.1	591	97
2001	6.2	597	96

For grains, factors such as the amino acid and protein content determine their usage in animal feeds (Pond and Maner 1984). For maximum benefit from the grains, animals should be fed the grains based on their requirement, use, and properties (Verstgen and van der Poel 2009). The grain intake should also correspond to the digestive capacity of the animals. Upon processing, the grains become more digestible (Rowe et al. 1999) and the rate of digestion changes. For instance, generally on feed consumption, starch digestion occurs in the rumen of the animals. By processing the grains, the degradation of starch in the rumen improves, increasing the utilization of the digested starch (Theurer 1986) by the ruminants. Therefore, processing of grains improves the feed utilization by animals.

### **2.5.1 Source of animal feed**

Conventional feeds have 40-50% grains (Dhakad et al. 2002). A high utilization of grains in feeds, increasing world population, and shortage of grains for human consumption has increased the cost of feeds considerably (Becker 2008). There is a need for alternative feeds that can lower feed prices (Dhakad et al. 2002). In addition, the optimal performance of the animals depends on the balanced diet, comprising of all essential nutrients. The nutrient requirement varies with the stage of growth of the animal. For example, with increasing stages of lactation, the amount of ruminally undegradable protein needed in dairy cows changes (NRC 1989). Similarly, the also varies as dietary fiber influences digestion and ruminal pH. The diet composition affects the dietary fiber requirement (Weiss et al. 1989). It also varies with the source of forage (Mertens 1983, as cited in Anderson and Schroeder 1999).

The NRC (1989) established standards for diets for different categories of animals. According to the standards, almost 25-28% NDF should be present in lactating dairy cow rations, out of which, 75% should be supplied by forages. This would prevent milk fat depression (Greco and Santos 2011), occurring at inadequate forage levels (Anderson and Schroeder 1999). Different feed ingredients can be provided to animals to achieve different goals. To improve the digestibility and feed intake, the ingredients having high fiber content should be selected, such as wheat middlings.

### **2.5.2 Low grain feeds**

The past decade has witnessed the shift from low grain to grainless diets (Dhakad et al. 2002). When grains were used commercially, they supplied most of the essential nutrients to livestock, pigs, sheep, and other ruminants. Grainless feeds were developed as an alternative to

reduce grain usage in animal feeds. Presently, deoiled rice bran and molasses (Malik et al. 1989, as cited in Dhakad et al. 2002) are used for buffalo calves, and wheat bran is used for adult sheep (Singh et al. 1999) and crossbred cattle calves (Mondal et al. 1996, as cited in Singh et al. 1999).

### **2.5.3 Wheat bran as animal feed**

Wheat bran has been used as animal feeds for decades. It provides the daily digestible fiber requirement of most of the ruminants. Dietary fibers resist breakdown in the alimentary canal, benefitting the animal. As a feed alternative, almost 50% wheat bran can replace barley in sheep diets (Singh et al. 1999) and corn in lamb diets (Dhakad et al. 2002). Wheat bran increases the nutritional value of feeds and prevents protein degradation in the rumen, thus improving animal performance. Similar findings were reported by Noblet and Goff (2001) for studies conducted on breeding sows.

### **2.5.4 Condensed distillers solubles (CDS) as animal feed**

Another alternate feed which has been under investigation is the CDS, a co-product of ethanol production. CDS has high fat and minerals values (Rausch and Belyea 2006; Kalscheur et al. 2008). It contains almost 20-30% crude protein (Lardy 2007), 1.4% phosphorus, 2.10% potassium, and 0.30% calcium, on a DM basis. Thin stillage, the substance evaporated to form CDS, is a well-known animal feed ingredient. Ojowi et al. (1996) analyzed wheat-based thin stillage as a nutrient supplement for cattle. The results of their experiments established the presence of high amounts of crude protein, and lower amounts of NDF, acid detergent fiber (ADF), and crude fat. They also stated that thin stillage could be used by livestock directly. Relative to wheat thin stillage, barley thin stillage was reported to have higher fiber, content and

lower protein content (Mustafa et al. 1999). The amino acid profile of barley-based thin stillage was better than wheat-based thin stillage as it contained more arginine, isoleucine, leucine, lysine, methionine, threonine, valine, aspartic acid, cysteine and serine. As an animal feed ingredient, CDS is a protein source for ruminants (Aines et al. 1985; NCRRP 1984), satisfying their minimal requirement of 15% protein.

CDS was used for citric acid production by Xie and West (2007) and provided animal health benefits. It prevented acidosis and improved feedlot performance (Lardy 2007), making the feed economical for animal diets (Kalscheur et al. 2008). Fumarate and malate were present in high amounts in the CDS (Fron et al. 1996) and they enhanced lactate fermentation and increased the growth of *Selenomona ruminantium* (Nisbet and Martin 1990, 1991, as cited in Fron et al. 1996). The CDS lactic acid content improved the rumen microflora activity, similar to distillers co-products (Ham et al. 1994), benefitting ruminant performance (Huntington and Britton 1979). Research conducted in later years also proved that by using CDS, cheese whey could be used to produce lactic acid (Liu et al. 2007). In certain cases, CDS is added back to distillers grains to form WDGS, and can be dried to form DDGS.

#### **2.5.5 Disadvantages of grain-based animal feed and grain-based ethanol co-products as feed ingredients**

Although grain-based feeds are increasingly being used as forage replacement, there are some disadvantages associated with them (Aines et al. 1986). Upon the consumption of grain-based feeds, starch digestion occurs in the rumen, which lowers the pH and reduces fiber digestion. As a result, the energy provided on feed consumption is lower than the predicted value. In terms of economy, there are some problems associated with grain-based feeds. With the



inclusion of almost 50% grains as feed ingredients, the cost of animal feed production has increased tremendously. Increasing demand for animal feeds have increased feed prices significantly, making grain-based feeds uneconomical. This has led to the increasing use of alternative feeds that can cut back feed production costs, without compromising on the nutritional requirement of animals. Similar to grain-based feeds, there are some disadvantages associated with grain-based ethanol co-products. For example, in using thin stillage, there are problems of transportation and storage. Likewise, distillers grains also have certain disadvantages. In the wet form, the water content of distillers grains limits the radius of transport, due to cost issues (Aines et al. 1986). Upon drying, the distillers grains can be conveyed to far-off areas. Still, it is less preferred as it is an expensive procedure, as drying (usually using rotary dryers) is an energy-intensive operation (Kingsly et al. 2010). Even though heat processing technologies such as drying increase P bioavailability, it has negative effect on the amino acids. Drying reduces the lysine content in DDGS, reducing the amino acid digestibility (Parsons et al. 2006). Over-heating of DDGS during the drying process increases the acid detergent insoluble crude protein (ADICP) in DDGS (Chen and Shurson 2004). ADICP is the heat damaged protein which is unavailable to the animals. Although the advantages associated with using thin stillage and distillers grains are sufficient to overlook these problems, more technological developments could help in overcoming these issues.

## **2.6 Processing of Animal Feed**

Every new technology is brought forth keeping in mind the advantages it would provide. Pelleting is one such technique having numerous advantages and is used in feed manufacturing. It is the process of compressing materials using heat and pressure at a certain moisture content

into a cylindrical form, known as pellet. Pelleting has been used extensively in the feed industry (Wood 1987). It is an expensive process (Skoch et al. 1983), but is being used commercially. The process cost is affected by the pellet quality, which in turn, is affected by the feed formulation (Thomas et al. 1998). The feed formulation also affects the pelleting process i.e. the throughput and energy consumption of the equipment.

### **2.6.1 Advantages of pelleting**

Pelleting of feed materials contributes to improved animal performance (Proudfoot and Sefton 1978). Pellets have better flowability (Briggs et al. 1999) which helps in their transportation and handling. During pelleting, changes in the functional components affect the physical properties of the animal feed pellets (Thomas et al. 1997). Wood (1987) also showed that protein denaturation affected pellet hardness. Thus, pelleting improves the physical properties of feed materials. Compaction of feeds also increases the bulk density of the materials (Fairfield 2003), reducing transportation costs. Pelleting is also beneficial in improving the feed intake of the animals. As more feed is consumed in the form of pellets, the daily nutrient intake improves, as the composition of pellets is fixed. Animals fed with feed pellets get a balanced diet, improving their production and growth. It ensures reduced selective feeding (Fairfield 2003). Pelleting also reduces feed wastage (Vanschoubrock et al. 1971) and improves process economics. Furthermore, pathogenic organisms are eradicated during the heat treatment, improving feed quality. In addition, thermal changes in starch and protein occur. Pelleting engages different types of feed ingredients (Behnke 1994, as cited in Fairfield 2003), reducing diet costs. Livestock producers attribute the improvement in animal performance to the different advantages of pelleting (Behnke 1998). With increased digestibility and reduced ingredient

segregation, pelleting improves the daily intake and, in turn, the animal performance is enhanced.

### **2.6.2 Pelleting process and pellet quality**

The pelleting process involves a series of operations including grinding the materials leading to particle size reduction, followed by conditioning, pelleting, and cooling of the materials (Thomas et al. 1997). The process can be simple or complicated involving multiple steps (Pipa and Frank 1989; Veenendaal 1990; van Zuilichem and van der Poel 1993). To ensure uniform distribution of ingredients, the feed materials are ground prior to pelleting. As a result of grinding, the surface area of the particles improve (Behnke 1996), reducing the process time. Ingredient mixing also affects the animal performance and pellet quality (Duncan 1973, as cited in Behnke 1996). Ruminant degradation is reduced by almost 15% (Tothi 2003) and the digestibility of DM and crude fiber is also affected (Thomas et al. 1997). In the feed pellets, starches and proteins are the primary ingredients (Wood 1987). By varying the process parameters, end products with differing functional properties are formed.

Even though the feed quality is essential, improvements in the physical quality is of primary importance. Transportation and handling damages from poorly formed pellets are undesirable (Robohm and Aspelt 1985; Fasina and Sokhansanj 1996 as cited Aarseth and Prestlokken 2003). Until consumed, pellets should remain intact (Crampton 1985, as cited in Aarseth and Prestlokken 2003). This implies that the pellets should be good in physical quality terms of hardness and durability. Addition of binders to feed ingredients during pellet production process can improve durability. Inter-particle contact and adhesive forces improve binding ability of feeds (Tabil et al. 1997). Heat processing and frictional heat generated when materials

pass through the die cause plasticizing of proteins and other changes improving the adhesive characteristics of binders. Studies by Wood (1987) revealed that the raw materials affected pellet quality and influenced commercial production. Moreover, reducing the microbial contamination in feeds (McCapes et al. 1989) by pelleting (Halls and Tallentire 1978) affects the hygienic quality of feeds and influences commercialization.

In order to optimize the pellet quality, the pelleting process should be controlled. Binder used, process parameters, and the raw material influence pellet quality (Thomas et al. 1999). The physical quality of pellets is analyzed by measuring pellet durability and hardness (Payne et al. 1994). Currently, broiler and turkey diets are pelleted (Behnke 1998) to improve the physical quality of feeds. Along with enhanced physical quality, weight gain and lower feed conversion has also been observed in pigs (Vanschoubroek et al. 1971; Pond and Maner 1984) and poultry (Moran 1989; Quemere et al. 1988; Calet 1965).

## **2.7 Summary**

Ethanol production and utilization is a globally booming industry, owing to increasing fuel demands and requirement of alternative fuels. As Canada joins this venture, wheat is the foremost choice for ethanol production owing to their availability and high yield capacity. Substantial ethanol production brings forth the need for technologies to enhance product and co-products value. Debranning ranks high among the various processes being used to improve the quality of ethanol. It enhances the efficiency of fuel ethanol plants by increasing the starch value in the feedstock. Essentially, the process should separate all the bran from the wheat kernels, with minimal starch loss. The debranning process has been done traditionally using frictional and abrasive equipment. Specifically, two abrasive mills, the Satake mill and the TADD, are

preferred for debranning because of their high debranning efficiency. Varying the grit size of the rollers, the retention time for the feed materials, and the speed of rotation of the rollers in the milling equipment are methods of improving debranning efficiency, which in turn, is positively related to ethanol production. As for the bran obtained on debranning of wheat, it has been utilized as a feed component for many years and its benefits as animal feed are well known. Another feed ingredient, the condensed distillers solubles (CDS), is also preferred for its feeding merits. The combination of wheat bran and CDS may further improve the quality of the commercially available feed. Additionally, improved feed quality will benefit the animals by improving the feed conversion efficiency. While aiming at improving the feed quality, technological developments in feed manufacture and feeding practices are the main focus. One of the highly recommended feed manufacturing processes is feed pelleting. Pelleting improves feed handling, storage, and palatability. Moreover, it improves the nutrient intake, influencing animal growth positively. Animal body weight gain accompanied with reduction in the feed fines represents feed production profits. These factors necessitate further research in pelleting animal feeds. This study aimed at the production of animal feed from ethanol co-products, namely wheat bran and CDS. Using these ingredients would improve the animal feed quality, without compromising on the ethanol production capacity and quality.

## **CHAPTER THREE**

### **STUDY 1: OPTIMIZATION OF THE DEBRANNING OF WHEAT USING LABORATORY EQUIPMENT FOR ETHANOL PRODUCTION**

#### **3.1 Abstract**

Ethanol production from starchy cereal grains is increasing rapidly due to increasing demand for alternative fuels. In Canada, wheat is the primary feedstock in ethanol plants. To improve the productivity of the ethanol plants in terms of product quality and yield, debranning of wheat grains may be employed. Debranning is advantageous in two ways. Firstly, bran removal increases the starch content of the feedstock, improving the fermentation efficiency of the ethanol plants. Secondly, bran, a valuable co-product can be used as an animal feed ingredient. In this study, experiments for bran removal were carried out using two abrasive equipment, the Satake and the TADD mill, to optimize the debranning process. Wheat samples (30 and 200 g) were debranned in the Satake mill at 1215, 1412, and 1515 rpm rotational speed, 30, 36, and 40 grit size, and 30, 60, and 90 s retention time, and in the TADD mill at 900 rpm rotational speed, 30, 36, 50, and 80 grit size, and 120, 180, 240, and 300 s retention time. In addition to debranning efficiency, the starch separation efficiency of the two mills was calculated at different debranning conditions. In the Satake mill, the 30 and 200 g sample size, 1412 and 1515 rpm rotational speeds, all grit sizes, and 60 s of retention time demonstrated the highest debranning efficiency. Correspondingly, optimal results in the TADD mill were obtained with 200 g sample size, 900 rpm rotational speed, 50 and 80 grit size, and 180 and 240 s retention time. However, based on the experimental results, Satake mill provided better debranning values compared to TADD mill. The starch separation efficiency values supported these results.

### **3.2 Introduction**

Ethanol production is a fast growing industry and has shown considerable impact on decreasing the world's reliance on fossil fuels. In order to maintain the growth of the ethanol industry, there is a need for sustainable supply of feedstock (Nixon 2009). Similar to the global scenario, ethanol production has increased tremendously in Canada, specifically in the Canadian prairies (Racz 2008). As a direct result of growth of the ethanol industry, the agricultural industry enjoys such benefits as job openings and more avenues of marketing for grain producers (Agriview 2007). Most of the ethanol plants in operation throughout Canada are grain-based. Therefore it is important that feedstock needed for ethanol production have sufficient availability. Inadequate corn production in the Canadian prairies and better economics on usage of locally grown wheat varieties makes wheat the first choice for ethanol production (Olar et al. 2004). Moreover, the quantity of wheat produced in Western Canada is sufficient for human and animal consumption, and as ethanol feedstock (Racz 2008).

With sufficient feedstock for utilization, the Canadian ethanol industry is bound to grow. A study by Licht (2005) showed that, in general, fuel ethanol production and consumption in Canada was low in the year 2004, which was only about 250 million L (Licht 2006). With increase in the economic and agricultural benefits associated with ethanol production, the ethanol production is expected to rise (Racz 2008). The availability of feedstock (Olar et al. 2004), job opportunities, and the local economic impact (Hooper 2011) were also some of the reasons for the increase in ethanol production. Also, high-grade wheat preferred for human consumption (varieties having low starch and high protein) is not being used in ethanol production (Saunders and Levin 2010). Instead, wheat varieties having high starch and low protein (Kindred et al.

2008) content are preferred. In 2010, ethanol production in Canada reached 1.7 billion L using wheat and 3.1 billion L using corn as feedstock, as shown by the survey results of Saunders and Levin (2010).

Due to increased availability of suitable (high starch, low protein) local wheat varieties, most of the ethanol processing plants in Canada use wheat as the primary feedstock. Almost 0.5 million t of wheat is used for manufacturing ethanol (Agriculture and Agri-Food Canada 2006). Wheat varieties suitable as ethanol feedstock often represent a good crop rotation fit, as reported by Reimer (2011). The ethanol industry also provides farmers with an additional local outlet for marketing the grain. Ethanol production provides the opportunity to use poor quality grain (affected by weather or disease) as feedstock (Bender 2011). The Canadian wheat varieties and their protein contents, as shown in Table 3.1, help in easier selection of feedstock for ethanol production. Wheat proteins contain the essential amino acid and lysine, and have better quality in terms of food and feed applications. High starch and low protein wheat varieties (Sosulski and Sosulski 1994) have appreciable starch conversion efficiency, and are preferred for ethanol production at the industrial level. The Canadian Prairie Spring, Canadian Western Red Winter, and Canadian Western Soft White wheat varieties are suitable for ethanol production [(S and T)<sup>2</sup> Consultants Inc. 2003].

High protein, low starch wheat is less preferred in ethanol production for three reasons. Firstly, the starch content in the grain should be high to be suitable for ethanol production (Sosulski and Sosulski 1994). Secondly, higher protein makes the mash adhesive, which is undesirable in ethanol production. Thirdly, poor solubility of wheat proteins affects the downstream processing of the spent grains (Agu et al. 2008). In addition, non-starch polysaccharides hinder processing by increasing the viscosity of the mash. The protein content of



soft white wheat is 8-9%, which is much closer to the protein levels of corn. Hard red spring wheat has higher protein content, and is less preferred. On the other hand, winter wheat is also a potential choice because it has yield higher than spring wheat varieties. The selected wheat varieties should be milled and processed for ethanol production.

Table 3.1 Canadian wheat varieties and their protein content

[(S and T)<sup>2</sup> Consultants Inc. 2003]

<b>Wheat class</b>	<b>Typical protein level (%- dry basis)</b>
Canadian Western Red Spring	13.2
Canadian Western Extra Strong	12.2
Canadian Prairie Spring Red	11.5
Canadian Western Red Winter	11.3
Canadian Prairie Spring White	11.2
Canadian Western Soft White	10.5
Canadian Western Amber Durum	12.8

Milling of grains, prior to fermentation, improves ethanol yield (Corredor et al. 2006). Pearling (debranning) was incorporated in commercial production of ethanol by Wang et al. (1997). Debranning uses friction and abrasion to partially remove the outer layers of the wheat kernel (Singh and Singh 2010) to further improve ethanol production. Debranning increases the starch content of the grains by removing the fiber and protein (Wang et al. 1997), thereby increasing the efficiency and yield of ethanol plants (Sosulski and Sosulski 1994). Wang et al. (1997) showed that dehulling increased the starch content of the debranned kernels by 12.2%. The ethanol yield per tonne of grain also increased by 6.5-22.5% (Sanchez and Cardona 2008),

thus increasing the production rate. Sosulski and Sosulski (1994) used the Allis-Chalmers mill for partial debranning of wheat. Complete debranning could not be done due to the presence of the crease on the ventral side of the kernel. The starch content of the whole grains was reported to be 54-57% and it was 64-68% in the debranned flour (Sosulski and Sosulski 1994).

Debranning using the Satake mill is highly beneficial. The mill is compact having characteristics such as good flowability and controlled processing (Dexter and Marchylo 2001). Another advantage of the Satake mill is its flexibility (Satake 1969). A flexible system adjusts the amount of bran removal, producing different products. Wang et al. (2007) studied dry oat dehulling using the Satake TM-05 abrasive mill. The results concluded that debranning removed the trichomes from the oats. In addition, high amount of pearled oat fractions, enriched in aleurone cells was produced.

The tangential abrasive dehulling device (TADD) is also a debranning equipment which works on the principle of abrasive dehulling. The TADD removes the bran layers using tangential abrasion (Hogan et al. 1964, as cited in Oomah et al. 1981) from grain kernels. The mill has been successfully used to debran different grains by Normand et al. (1965). Upon usage, it was seen that at higher retention times, the bran fraction versus retention time plot in TADD was nonlinear (Lawton and Faubion 1989). Since the Satake and the TADD mill have been used successfully for dehulling grains, it is essential to optimize the mill operations during debranning of wheat when it is considered as a feedstock preparation process prior to ethanol fermentation. Optimal conditions of debranning will lead to improvement in the quality of the feedstock and the final product.

The objective of this study was to optimize the laboratory debranning process of wheat using the two milling equipment, the Satake mill and the tangential abrasive dehulling device (TADD), before using it as feedstock for ethanol production.

### **3.3 Materials and Methods**

#### **3.3.1 Materials**

The wheat grain (AC Andrew, a soft white spring variety) used in this study was obtained from an ethanol plant located in southern Saskatchewan, Canada. They were stored in large plastic bins to protect the grain from rodent infestation. A dockage tester (Style no. C-XT3, Cea-Simon-Day Ltd., Winnipeg, MB) was used to mechanically separate the various components i.e. broken kernels, stones, and other grains present among the wheat grains, based on their weight and particle size (Opoku et al. 2003a). The machine was turned on and the sample was discharged into the hopper. The materials were collected after they passed the hopper and the sieves. The sieves were tapped for this purpose. On completion of the test, the machine was turned off. Based on their particle size, the materials were poured into separate pans. Only clean whole wheat grains were used for dehulling.

#### **3.3.2 Experimental methods**

Two types of abrasive mills, the Satake mill and the TADD, were used to conduct optimization studies for bran production. For the optimizing process, the sample size, the rotational speed, the retention time, and the grit size (nominal size of abrasive particles corresponding to the number of openings per inch in a screen through which the particles can

pass) were varied in the two mills. Each test was conducted with three replicates for reproducibility of results.

### **3.3.2.1 Experimental plan**

The experimental plan followed is given in Table A.1 in the Appendix. Wheat samples (30 and 200 g) were debranned in the Satake mill at rotational speeds of 1215, 1412, and 1515rpm, grit sizes of 30, 36, and 40, and retention times of 30, 60, and 90 s and in the TADD mill at rotational speed of 900 rpm, grit sizes of 30, 36, 50, and 80, and retention times of 120, 180, 240, and 300 s. The starch content of the debranned kernels was used to calculate the separation efficiency of the debranning equipment.

### **3.3.2.2 Dehulling procedure**

Dehulling in Satake mill - The dehulling experiment was done in the Satake mill (Satake Grain Testing Mill Model TM-05 Satake Engineering Co., Ltd, Japan; Figure 3.1). The wheat obtained running through the dockage tester ready for dehulling. The feed hopper, placed on vertically on the top of the mill was used to pour the grains into the milling chamber. On entering the abrasion chamber, the roller and the metallic casting dehulled the grains (Opoku et al. 2003a). The abraded grains moved to the discharge end. There was friction between the kernels and the screen, completing the debranning process. When milling was completed, the wooden box attached to the milling chamber contained the husk and bran (Opoku et al. 2003a), and the grains were collected separately. The degree of debranning was controlled by various factors. For this research, 30 and 200 g of wheat were used for each test, and the sample size, rotational speed, grit size, and retention time were the factors to be optimized.



Figure 3.1 The Satake mill

Dehulling in the Tangential abrasive dehulling device (TADD) - Dehulling of wheat was also done in the TADD mill (Model no. 4E-230, Venables Machine Works, Saskatoon, SK; Figure 3.2). The mill had two main components: a horizontally placed roller and a stationary aluminum head plate holding eight stainless steel sample cups (Opoku et al. 2003b; Figure 3.3). Usually, for each run, any two opposing open bottomed sample cups are used to place the grain samples/grains. Each test had a fixed residence time, maintained by a digital electronic timer (Model 8683-10, ColeParmer Instrument Company, Chicago, IL; Opoku et al. 2003b). On completion of debranning, a vacuum aspirator was used to collect the debranned grains (Oomah et al. 1981), whereas a cyclone separator device connected to the TADD collected the husk and bran separately (Opoku et al. 2003a). Sample sizes of 30 and 200 g were used for each test in this research, and the sample size, grit size, and retention time were the factors to be optimized. Rotational speed had been optimized earlier and the optimal value (900 rpm) was used throughout the experiments.

### **3.3.2.3 Moisture content determination:**

Moisture content (wet basis) of whole wheat and wheat bran samples obtained after debranning was determined using the air-oven method (AOAC Method 930.15, AACC 1995). The moisture content was analyzed by drying 2-3 g of the samples (ground) at 135°C for 2 h. The moisture content was expressed as the percentage of the total mass, i.e. mass of water/initial mass of the sample. Three runs were conducted for the samples from the two abrasive devices.



Figure 3.2 The tangential abrasive dehulling device

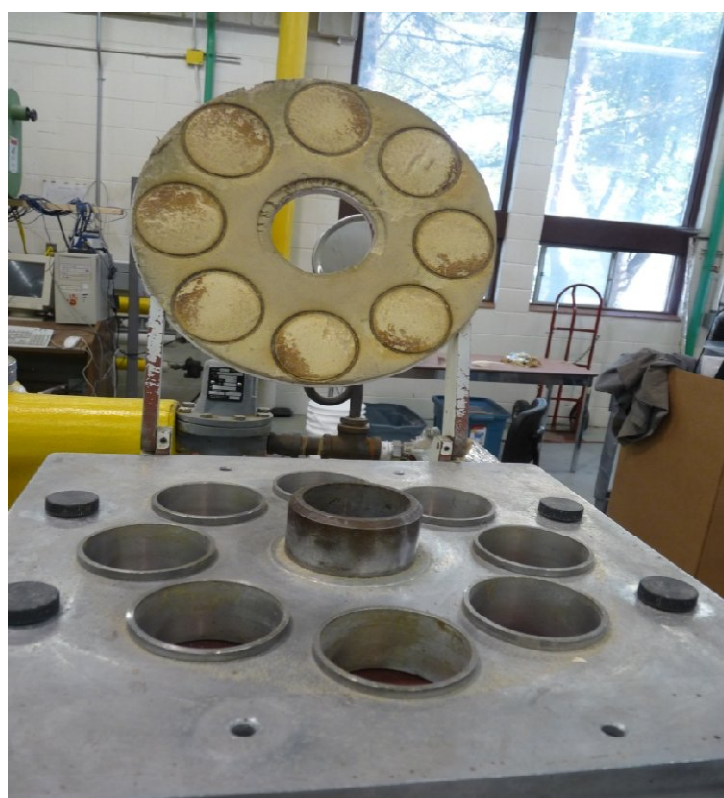


Figure 3.3 The sample cup arrangement in the TADD

#### 3.3.2.4 Starch content and starch separation efficiency

Prior to starch content analysis, the Satake and the TADD mill debranning results were compared. The combinations of the three machine variables i.e. speed, grit size, and retention time, leading to optimal bran production were selected and only those combinations were further analyzed for starch content. The AOAC (Method 996.11, AOAC 1998) and AACC (Method 76.13, AACC 2000) methods are a simple and reliable procedure for the measurement of total starch.

The starch separation efficiency for the two abrasive devices, the Satake mill and the TADD was calculated by analyzing the debranned wheat obtained from the devices for the starch content. Starch analysis was carried out by following the procedure as stated in AOAC Method 996.11 and AACC Method 76.13 for total starch assay (Amyloglucosidase/  $\alpha$ -Amylase method; McCleary et al. 1994). Each sample was replicated thrice. The percentage of the total starch retained in the debranned kernel that was recovered is a measure of the starch separation efficiency of the debranning equipment. The starch separation efficiency for the two mills was calculated according to the formula used by Tyler et al. (1981) which is given below.

$$\text{Starch separation efficiency} = \left( \frac{\text{wt. of debranned wheat} * \text{total starch in debranned kernels}}{\text{wt. of whole wheat} * \text{total starch in whole wheat}} \right) * 100$$

#### 3.3.2.5 Data analysis

Statistical analysis of the results obtained for debranning and starch analysis were done using SAS Version 9.2 Software (SAS 2008). The GLM and ANOVA procedures were used in the Student-Newman-Keuls test. Analysis of variance was used to determine if differences



between treatments were significant at 5% significance level and to determine the relationship between the parameters of debranning (rotational speed, grit size, and retention time) in the Satake mill and the TADD.

### **3.4 Results and Discussion**

Experiments were conducted to determine the effect of variation in sample size, rotational speed, grit size, and retention time on the debranning of wheat and starch separation efficiency of the Satake and the TADD mill. The comparison in the debranning efficiency of the Satake and TADD mill is based on the mechanisms of abrasion.

#### **3.4.1 Debranning**

##### **3.4.1.1 Debranning in the Satake mill (30 g sample size)**

Debranning efficiency of a mill is an important machine characteristic. Studies by Wang (2005) had reported that dehulling of grains in the Satake mill is affected by the process variables such as rotational speed and dehulling time. The results for debranning with different sample sizes (30 and 200 g) for the Satake mill are given in Table 3.2 and Table 3.3 respectively. The amount of bran fraction obtained is an indicator of debranning efficiency of the abrasive mill.

The results from Table 3.2 indicate that for a sample size of 30 g and 1215 rpm rotational speed, the highest amount of bran fraction produced was 11.02% (grit size-36, retention time-90 s). The bran fractions for all the three grit sizes with a lower retention time (30 and 60s) were less than 7%. Studies by Oomah et al. (1981) and Mbengue (1986, as cited in Bassey and Schmidt 1989), have emphasized that factors such as grit size of the grinding surface affect the dehuller performance.

Table 3.2 Debranning of wheat (AC Andrew) using the Satake mill (30 g)

Sample size (g)	Rotation speed(rpm)	Grit size	Retention time (s)	Bran (%)*
30	1215	30	30	2.83 (0.05)a
30	1215	30	60	6.81 (0.41)b
30	1215	30	90	10.82 (0.75)c
30	1215	36	30	2.73 (0.22)a
30	1215	36	60	6.17 (0.26)b
30	1215	36	90	11.02 (0.53)c
30	1215	40	30	2.51 (0.11)a
30	1215	40	60	6.53 (0.08)b
30	1215	40	90	10.89 (0.75)c
30	1412	30	30	5.39 (0.44)a
30	1412	30	60	12.65 (0.15)b
30	1412	30	90	20.29 (0.37)c
30	1412	36	30	5.19 (0.42)a
30	1412	36	60	12.01 (0.46)b
30	1412	36	90	19.21 (0.30)c
30	1412	40	30	5.37 (0.38)a
30	1412	40	60	11.72 (0.16)b
30	1412	40	90	18.78 (0.21)c
30	1515	30	30	7.78 (0.15)a
30	1515	30	60	17.31 (0.46)b
30	1515	30	90	26.30 (0.67)c
30	1515	36	30	7.37 (0.34)a
30	1515	36	60	15.19 (0.52)b
30	1515	36	90	23.07 (0.49)c
30	1515	40	30	5.86 (0.33)a
30	1515	40	60	14.47 (0.62)b
30	1515	40	90	23.59 (0.38)c

Number in parenthesis is standard deviation, \*n = 3; a, b, c = indicate that means with the same letter in a column are not significantly different at P = 0.05

Results of the experiments of this study also lead to similar conclusions. Higher rotational speeds (1412 and 1515 rpm) and different combinations of grit size and retention time were able to cause extensive bran removal. For the rotational speed of 1515 rpm, the three grit sizes (30, 36, and 40) and a 90 s retention time lead to removal of a very high bran fraction (26.30%, 23.07%, and 23.59% respectively), which was beyond the desired optimal value. The combination for 30 g sample size which lead to optimal bran removal were 1412 rpm and 1515 rpm, all grit sizes (30, 36, and 40), and 60 s retention time. For the wheat grain, there was an increase in bran fraction with increasing rotational speed. This was similar to the trend followed by oat grains dehulled in an impact dehuller (Peltonen-Sainio et al. 2004). Statistical analysis for Satake mill (Table A.2) showed that the three variables (rotational speed, grit size, and retention time) had positively significant effect on debranning ( $P < 0.01$  for each). Also, the interaction between speed and grit size as well as speed and retention time was positively significant ( $P < 0.01$  for each).

#### **3.4.1.2 Debranning in the Satake mill (200 g sample size)**

Using the 200 g sample size, the rotational speed of 1215 rpm was optimal for 12-18% bran fraction production (Table 3.3). The results were consistent with the findings of McCluggage (1943, as cited in Liu 2007), who stated that the degree of dehulling increased as mass of the grain in the dehuller increased. The debranning results for the Satake mill for 200 g sample size (30, 36, and 40 grit sizes) also indicated that using higher retention time (90 s) resulted in removal of very high amount of bran from the wheat grains (30-38%). For 200 g sample size, the three rotational speeds (1215, 1412, and 1515 rpm) and 60 s retention time provided optimal results.

Table 3.3 Debranning of wheat using the Satake mill (200 g)

Sample size (g)	Rotation speed(rpm)	Grit size	Retention time (s)	Bran (%)*
200	1215	30	30	3.63 (0.09)a
200	1215	30	60	17.11 (0.56)b
200	1215	30	90	31.94 (0.23)c
200	1215	36	30	4.44 (0.83)a
200	1215	36	60	18.06 (0.12)b
200	1215	36	90	33.05 (0.53)c
200	1215	40	30	6.69 (0.05)a
200	1215	40	60	18.19 (0.26)b
200	1215	40	90	30.64 (0.06)c
200	1412	30	30	5.65 (0.39)a
200	1412	30	60	20.03 (1.16)b
200	1412	30	90	35.73 (0.86)c
200	1412	36	30	6.52 (0.11)a
200	1412	36	60	21.65 (1.13)b
200	1412	36	90	36.01 (0.19)c
200	1412	40	30	5.65 (0.31)a
200	1412	40	60	18.52 (0.76)b
200	1412	40	90	33.21 (0.32)c
200	1515	30	30	6.08 (0.98)a
200	1515	30	60	14.03 (0.26)b
200	1515	30	90	31.83 (0.29)c
200	1515	36	30	6.75 (0.11)a
200	1515	36	60	20.82 (0.32)b
200	1515	36	90	37.85 (0.69)c
200	1515	40	30	4.17 (0.10)a
200	1515	40	60	22.90 (0.02)b
200	1515	40	90	36.60 (0.34)c

Number in parenthesis is standard deviation, \*n = 3; a, b, c = indicate that means with the same letter in a column are not significantly different at P = 0.05

Similar to the 30 g sample size, with a constant retention time and increasing rotational speed of the mill, there was increased bran removal as the grit size increased. Statistical analysis for the Satake mill (200 g sample size, Table A.3) showed that the three variables (rotational speed, grit size, and retention time) had positively significant effect on debranning ( $P < 0.01$  for each). Although all the three variables were significant, retention time had the highest effect on debranning efficiency. Also, the interaction among the three variables was positively significant ( $P = 0.02$ ).

The debranning results from the Satake mill (30 g and 200 g) showed that optimal bran fraction values could be obtained with both sample sizes, but with varying speed, grit size, and retention time. For both sample sizes, 90 s retention time lead to removal of more than 20% bran. This indicates that along with the bran, outer endosperm layers were also being removed during the debranning process. This would lead to an undesirable loss of starch from the debranned kernels. As a result, the starch content of the debranned wheat kernels being used as feedstock for ethanol production would be reduced, lowering the quality of the final product.

#### **3.4.1.3 Debranning in the TADD mill (30 g sample size)**

The debranning results for the TADD mill for the two sample sizes (30 g and 200 g), rotational speed (900 rpm), grit sizes (30, 36, 50, and 80), and retention times (120, 180, 240, and 300 s) are given in Tables 3.4 and 3.5. The results showed that the degree of debranning was regulated by grit size, retention time, and other factors. The results of the experiments had conclusions similar to the results reported by Posner and Hibbs (1997) on wheat flour milling. Their study revealed that grit roughness and roller speed affected debranning efficiency. With 30 g as the sample size and at 900 rpm rotational speed, on varying the grit size and increasing the

retention time, the percent bran fraction obtained increased. This implies that under each combination of rotational speed and grit size, on increasing the retention time, there was a slight increase in the bran fraction removal. Similar conclusions were drawn by Mwasaru et al. (1988) who stated that abrasive surface of the TADD mill affected dehulling efficiency. Furthermore, under each combination, the bran fraction removal was much lower than the optimal value. The highest bran fraction percentage achieved with 30 g sample size was 7.28%, which was attained by using 900 rpm speed, 80 grit size, and 300 s retention time.

Table 3.4 Debranning of wheat using the tangential abrasive dehulling device (TADD, 30 g)

<b>Sample size (g)</b>	<b>Rotation speed (rpm)</b>	<b>Grit size</b>	<b>Retention time (s)</b>	<b>Bran (%)*</b>
30	900	30	120	1.09 (0.09)a
30	900	30	180	2.54 (0.36)b
30	900	30	240	3.89 (0.28)c
30	900	30	300	4.35 (0.13)d
30	900	36	120	2.00 (0.08)a
30	900	36	180	2.94 (0.04)b
30	900	36	240	3.89 (0.21)c
30	900	36	300	4.93 (0.03)d
30	900	50	120	2.47 (0.03)a
30	900	50	180	3.35 (0.08)b
30	900	50	240	4.80 (0.22)c
30	900	50	300	6.36 (0.01)d
30	900	80	120	3.22 (0.20)a
30	900	80	180	4.01 (0.06)b
30	900	80	240	5.40 (0.08)c
30	900	80	300	7.28 (0.12)d

Number in parenthesis is standard deviation, \*n = 3 a, b, c = indicate that means with the same letter in a column are not significantly different at P = 0.05

The statistical analysis results for 30 g sample size (Table A.4) showed that both grit size and retention time had positively significant effect on debranning efficiency of the mill ( $P < 0.01$  for both). Variable interaction was insignificant.

#### **3.4.1.4 Debranning in the TADD mill (200 g sample size)**

McCluggage (1943, as cited in Lawton and Faubion 1989) stated that the quantity of sample per run and the dimensions of the grains influenced milling. According to the experiments done in this study, increasing the sample size to 200 g gave better debranning results. The results showed that with a constant rotational speed, usage of higher grit size was preferable. With 50 and 80 grit size and 180, 240, and 300 s retention time, the bran fraction obtained ranged from 13–21%. Upon using 50 and 80 grit size, and 180 and 240 s retention time, optimal debranning could be attained. The statistical analysis results for 200 g samples size (Table A.5) suggested that retention time and grit size were significant factors affecting debranning efficiency positively ( $P < 0.01$  for both). In contrast to the results for 30 g sample size, grit size and retention time interaction had positively significant effect on the bran fraction ( $P = 0.03$ ).

Debranning in the TADD resulted in lower yield of bran fraction under the above conditions as compared to the Satake mill. The debranning results for 30 g sample size were lower than the optimal range (12-18% bran fraction) which is unacceptable. In the case of 200 g sample size, the optimal range for bran fraction was attained at 50 and 80 grit size and 180 and 240 s retention time, providing values comparable to the Satake mill results. In their evaluation of the application of the TADD mill, Reichert et al. (1986) observed a linear relationship

between disc speed and dehulling efficiency. Since the rotational speed was kept constant for both sample sizes in this study, this aspect could not be analyzed.

Overall, the Satake mill indicated maximized dehulling efficiency. Although the TADD mill at 200 g sample size was able to provide results of debranning comparable to the Satake mill results, it was also noted that the time for debranning in the TADD mill was much longer than the Satake mill. While the Satake mill reached optimal debranning values at 60 s, it took 240-300 s in the TADD mill. Moreover, the Satake mill and TADD differ in the mechanics of abrasion. The Satake mill has a vertically rotating abrasive roller which provides more abrasive surface for debranning. As the materials entered the abrasion chamber, they were continuously mixed, improving the rate of debranning and reducing the retention time. In the TADD, the horizontally placed disc and the sample cups provided lesser abrasive surface. The sample size per sample cup was also a limiting factor. Abrasion in the TADD mill took longer, decreasing the production rates. On comparing the Satake mill and TADD, the abrasion mechanism used in the Satake mill is preferable for industrial processes. Debranning equipment with similar mode of action as the Satake mill would increase the production rates.

For similar reasons, the Satake mill was used by Wang (2005) to dehull red lentils and by Black et al. (1998) to dehull field peas. The results from the TADD and Satake mill demonstrated that the milling conditions impact the dehulling characteristics of the grains, which was also the conclusion drawn by Wang (2005).



Table 3.5 Debranning of wheat using the tangential abrasive dehulling device (TADD, 200 g)

Sample size (g)	Rotation speed (rpm)	Grit size	Retention time (s)	Bran (%)*
200	900	30	120	3.53 (0.30)a
200	900	30	180	3.99 (0.37)a
200	900	30	240	5.63 (0.74)b
200	900	30	300	7.10 (0.15)c
200	900	36	120	3.19 (0.13)a
200	900	36	180	5.18 (0.21)a
200	900	36	240	6.61 (0.01)b
200	900	36	300	8.13 (0.36)c
200	900	50	120	9.17 (0.40)a
200	900	50	180	13.43 (0.53)b
200	900	50	240	16.38 (0.64)c
200	900	50	300	18.61 (0.57)d
200	900	80	120	10.78 (0.37)a
200	900	80	180	16.01 (0.07)b
200	900	80	240	19.94 (0.09)c
200	900	80	300	21.51 (0.09)d

Number in parenthesis is standard deviation, \*n = 3; a, b, c, d = indicate that means with the same letter in a column pare not significantly different at P = 0.05

### 3.4.2 Starch content

Sosulski and Sosulski (1994) used partial debranning method to improve the ethanol yield from barley, rye, and triticale. In their experiment, the starch content increased from 54-57% in the whole grains to 64-68% in the flours by debranning. This study also aimed at similar results.

Moisture content of grains is an important factor which affects the physical properties of grains in various ways. Previous studies have reported that moisture content is required for starch gelatinization (Moritz et al. 2002). Dziki (2004) reported that the rupture point on the application

of force in the wheat grains varied with the moisture content. Furthermore, wheat grain moisture content influences grain hardness (Katz et al. 1961) which in turn affects debranning (Obuchowski and Bushuk 1980). According to Dziki and Laskowski (2005), when the debranning of grains precedes grinding, the power required for the process is lower, making it economical. Considering these aspects, the starch analysis of the debranned kernels was done to evaluate the separation efficiency of the debranning equipment.

#### **3.4.2.1 Starch content of the debranned kernels from the Satake and the TADD mill (30 g sample size)**

The samples from the debranning test which provided optimal bran fraction were selected for starch analysis. The starch content of the debranned kernels from the Satake mill and the TADD (30 g sample size) are shown in Table 3.6. For the Satake mill samples, the starch content of the kernels increased on debranning by almost 8.0% (from 70.4% to 76.0%). According to Wang (2008), the starch content of grains and the debranning efficiency were directly proportional. El Hag et al. (2002) also stated that dehulling significantly increased the starch content in the grains. Since the bran contains very little starch, its removal means that there is proportionally more starch in the dehulled grains. Rios et al. (2009) concluded that dehulling isolated the starchy endosperm and reduced fungal contaminations in wheat flours. Therefore, optimal debranning results were an indicator of less starch loss from the grains.

Starch separation efficiency of the Satake mill varied from 67 to 91%, depending upon the starch content of the debranned wheat samples. The TADD mill sample (bran fraction 7.28%) used for starch analysis was obtained with the rotational speed of 900 rpm, 80 mm grit size, and 300 s retention time. The starch content of the sample (78.8%) was higher than the

Satake mill samples (Table 3.6), owing to partial or incomplete debranning. Wang et al. (1997) demonstrated that the starch content of the dehulled grains increased as the bran layers were removed progressively. The grains might have undergone partial debranning in the TADD mill and some of the starch from the inner bran layers might be retained with the grain kernels. According to the equation formulated by Tyler et al. (1981), the starch separation efficiency was positively correlated to the starch content of debranned grains. For starch content, the process variables had a positively significant effect. The grit size had the highest effect ( $P < 0.01$ ), whereas rotational speed and retention time had a lower effect ( $P = 0.02$  and  $0.01$ , respectively [Table A.6]). The interaction between the variables was low or insignificant ( $P > 0.05$ ). In case of starch separation efficiency of the Satake mill, rotational speed, grit size, and the interaction between them was positively significant ( $P = 0.02$ ,  $0.02$ , and  $0.03$  respectively [Table A.7]). Since only one TADD mill sample was acceptable for starch analysis, no statistical analysis could be done.

From the results obtained for 30 g sample size, the optimal condition for debranning in the Satake mill was determined to be 1515 rpm rotational speed, 40 grit size, and 60 s retention time. The TADD mill results were poor compared to the Satake mill, with lower debranning and starch separation efficiency.

Table 3.6 Starch content of the debranned kernels from the Satake mill and the TADD (30 g)

Sample	Sample size (g)	Speed (rpm)	Grit size	Retention time (s)	Moisture content (%w.b)*	Starch content (%)* (kernel)	Starch separation efficiency (%)
Initial wheat grains		—	—	—	11.87	70.4 (3.1)	—
Satake	30	1412	30	90	9.28a	72.6a (3.0)	70.0a
Satake	30	1412	36	60	9.48a	76.2a (0.9)	69.7a
Satake	30	1412	40	90	9.83a	75.5a (1.4)	67.5a
Satake	30	1515	30	60	10.19a	60.3a (4.9)	71.4a
Satake	30	1515	30	90	9.74a	68.6a (0.9)	71.6a
Satake	30	1515	36	60	10.21a	71.7a (2.1)	88.1a
Satake	30	1515	36	90	10.36a	75.5a (2.0)	83.4a
Satake	30	1515	40	60	9.92a	74.4a (1.0)	91.7a
Satake	30	1515	40	90	9.81a	76.2a (1.0)	84.5a
TADD	30	900	80	300	8.24	78.8 (1.0)	101.2

Number in parenthesis is standard deviation, \*n = 3, a = indicate that means in a column are not significantly different at P = 0.05

### **3.4.2.2 Starch content of the debranned kernels from the Satake mill (200 g sample size)**

Debranned kernel samples from the Satake mill were tested for starch content and the results of starch analysis are given in Table 3.7. The results indicated that sample size of 200 g could be highly recommended because the starch separation efficiency achieved under varying conditions went up to 98%. The data for starch content of the debranned kernels showed that rotational speeds 1412 and 1515 rpm, grit sizes 30, 36, and 40, and retention time 90 s gave the highest values (more than 85%). From the starch separation efficiency formula, it is understood that separation efficiency is directly proportional to the debranned kernel weight and the starch content of the debranned kernels. Also, starch content is inversely proportional to kernel weight i.e. as complete debranning occurs, kernel weight reduces and the overall starch content in the kernel increases. Thus, with a decrease in kernel weight and an increase in starch content of kernels, the separation efficiency is affected both ways. The general trend in the data obtained was that, as grain kernel weight decreased (higher retention time in debranning), the starch content increased while the separation efficiency decreased.

The results from the starch analysis showed that rotational speeds 1412 and 1515 rpm, 40 grit size, and 90 s retention time lead to higher starch content in debranned kernels (~ 93%). Also, with the same rotational speeds, all the grit sizes of the Satake mill caused high starch separation with 60 s retention time (more than 85%). For the starch content, retention time had the highest positively significant effect ( $P < 0.01$ , Table A.8). The effect of rotational speed on the starch content was also positively significant ( $P < 0.01$ ) whereas interaction between the two and grit size had a lower significance ( $P = 0.03$  for both).

Table 3.7 Starch content of the debranned kernels from the Satake mill (200 g)

Speed (rpm)	Grit size (mm)	Retention time (s)	Moisture content* (%)	Starch content* (%)	Starch separation efficiency** (%)
1215	30	30	9.29a	64.3(1.0)a	89.3(1.3)a
1215	30	60	9.46b	68.8(0.6)a	80.6(0.8)a
1215	30	90	9.81c	71.6(1.5)a	68.7(1.5)b
1215	36	30	9.28a	52.1(2.5)a	71.8(3.5)a
1215	36	60	9.48b	63.4(0.6)a	73.5(0.7)a
1215	36	90	9.8c	71.8(0.1)a	67.8(0.1)b
1215	40	30	9.3a	65.3(1.7)a	87.0(2.2)a
1215	40	60	9.47b	75.4(0.2)a	88.1(0.3)a
1215	40	90	9.85c	85.0(1.7)a	83.1(1.7)b
1412	30	30	9.26a	69.0(0.6)a	94.4(0.8)a
1412	30	60	9.52b	75.2(3.6)b	85.0(4.1)a
1412	30	90	10.1c	87.7(0.1)c	79.4(0.1)a
1412	36	30	9.19a	75.3(0.9)a	99.0(1.2)a
1412	36	60	9.51b	83.0(1.7)b	92.0(1.8)a
1412	36	90	9.81c	90.1(0.6)c	80.6(0.5)a
1412	40	30	9.28a	65.8(0.2)a	89.9(0.2)a

Speed (rpm)	Grit size (mm)	Retention time (s)	Moisture content* (%)	Starch content* (%)	Starch separation efficiency** (%)
1412	40	90	9.84c	93.6(0.8)c	87.6(0.7)a
1515	30	30	9.84a	63.9(2.0)a	86.2(2.8)a
1515	30	60	10.19a	78.8(1.1)b	96.6(1.4)a
1515	30	90	9.74a	89.8(1.2)c	86.5(1.2)a
1515	36	30	10.78a	67.9(0.9)a	91.6(1.3)a
1515	36	60	10.21a	80.6(1.8)b	91.0(2.0)a
1515	36	90	10.36a	89.4(0.2)c	77.3(0.2)a
1515	40	30	9.66a	64.1(0.7)a	88.4(0.9)a
1515	40	60	9.92a	78.3(0.3)b	86.6(0.4)a
1515	40	90	9.81a	93.0(0.8)c	84.7(0.7)a

Number in parenthesis is standard deviation, \*n = 3, \*\*initial whole wheat starch content = 69.0%; a, b, c = indicate that means with the same letter in a column are not significantly different at P = 0.05

In the case of starch separation efficiency of the Satake mill, rotational speed and retention time held high positive significance ( $P < 0.01$ , Table A.9). The interaction between the variables and the effect of grit size were insignificant ( $P > 0.05$ ).

### 3.4.2.3 Starch content of the debranned kernels from the TADD mill (200 g sample size)

Four of the samples from the TADD mill gave optimal debranning values and were selected for starch analysis. From the results obtained (Table 3.8), it was seen that the starch content of the debranned kernels increased up to 68% owing to incomplete debranning. Similarly, starch separation efficiency of the TADD mill was only as high as 80% under 900 rpm rotational speed, 50 grit size, and 300 s retention time. This indicated that the TADD mill had lower efficiency of debranning and starch separation as compared to the Satake mill. The statistical analysis results revealed that both grit size and retention time (process variables) positively affected starch content of the debranned kernels and the separation efficiency of the TADD mill. The P-values for both were higher than 0.05 (Table A.10).

Table 3.8: Starch content of the debranned kernels from the TADD mill (200 g)

Speed (rpm)	Grit size (mm)	Retention time (s)	Moisture content* (%)	Starch content* (%)	Starch separation efficiency** (%)
900	50	180	10.12	53.6(0.3)a	67.2(0.3)a
900	50	240	10.05	64.4(0.3)a	78.0(0.3)a
900	50	300	9.86	68.0(2.9)a	80.2(3.4)a
900	80	180	9.98	61.2(1.8)a	74.4(2.2)a

Number in parenthesis is standard deviation, \*n = 3, \*\*initial whole wheat starch content = 69.0%; a = indicate that means in a column are not significantly different at  $P = 0.05$



### **3.5 Summary and Conclusion**

The experiments conducted helped in optimizing the two laboratory abrasive mills, the Satake and the TADD mill. The first sample size used for the debranning experiments was 30 g. The results showed that on using 30 g sample size, the debranning efficiency of both the mills was low. A lower debranning efficiency would reduce the productivity of the mills. Studies have shown that larger sample sizes improve abrasion accuracy. Two hundred grams was the maximum applicable sample size suggested in previous studies. Due to this reason, 200 g was the second sample size used in this study to improve the debranning efficiency. The optimal conditions of debranning in Satake mill were found to be 200 g sample size, 1412 and 1515 rpm rotational speeds, all grit sizes, and 60 s retention time. In the case of TADD, 200 g sample size, 900 rpm rotational speed, 50 and 80 grit sizes, and 180 and 240 s retention time provided results slightly comparable to the Satake results. The experimental results concluded that the Satake mill provided better debranning results as compared to the TADD. This is because, on using the Satake mill, optimum bran production could be done in 60 s whereas in case of the TADD mill, it took at least 240 s. This indicated that the Satake mill had higher productivity. The results of starch separation efficiency showed that the Satake mill was more preferable compared to the TADD. The results indicated that sample size of 200 g, rotational speed of 1515 rpm, 30 grit size, and 60 s retention time are the optimum condition for bran production for the Satake mill. Similarly, 200 g sample size, 900 rpm rotational speed, 50 grit size, and 240 s retention time are the optimum conditions for the TADD mill.

## **CHAPTER FOUR**

### **STUDY 2: PELLETING CHARACTERISTICS OF BRAN AND CONDENSED DISTILLERS SOLUBLES (CDS) AS ANIMAL FEED**

#### **4.1 Abstract**

The rapid growth of grain-based ethanol plants has increased the amount of co-product available as feed ingredients. Based on the increased feed demands and fluctuations in commodity prices, wheat-based ethanol co-products are being incorporated as feeds. For this study, two of the co-products, namely, wheat bran and condensed distillers solubles (CDS), were blended in three varying proportions (90% bran:10% CDS, 80% bran:20% CDS, and 70% bran:30% CDS) for production of highly durable feed pellets. The moisture content of the bran-CDS mash was lowered to 12-14% by drying in the heat pump assisted cabinet dryer (35°C and 40% RH) to ensure pelleting of the materials. The experiments were split into two stages. First stage experiments used the single pelleting unit to optimize the pelleting process variables. Second stage experiments used the pilot-scale pellet mill to verify the optimized variables of the single pelleting unit. The pellets were stored at a controlled environment for 14 d. Tests for the physical properties, such as durability, density, dimensional stability, and moisture content were conducted after cooling the pellets in ambient temperatures and after 14 d of storage. The single-pelleting results indicated that bran-CDS ratio and pelleting temperature affected the physical quality of the pellets produced. Under high pelleting temperatures (90°C), pellets with durability of more than 95% were produced. Specifically, the 90:10 bran-CDS ratio produced pellets of high durability and dimensional stability at high pelleting temperatures. The pilot-scale mill produced highly durable feed pellets for 70:30 and 90:10 bran-CDS ratios when similar pelleting

conditions were provided. After cooling, the physical characteristics of the 70:30 ratio improved further. Moreover, a better nutritional profile made the 70:30 ratio the preferred animal feed with good characteristics.

## **4.2 Introduction**

Animal feed utilizes almost 50% of the grains produced globally (Sansoucy 1995). Increased feed demands and consumption of animal-based food products have acted as catalysts (Sapkota et al. 2007) for animal feed production. In addition, commercially available animal feeds, composed of animal wastes (Rhodes and Orton 1975) and animal by-products, may be hazardous causing human food contamination (Sapkota et al. 2007). For these reasons, there is dire need of improvement in the animal feeding practices and feed processing technologies. Feed manufacturers are compelled to generate high quality feeds, to satisfy the requirements of different breeds and to ensure improved animal performance (Behnke 1996). Other than animal feeds, grains are also utilized for production of high quality ethanol.

Whole grains such as corn, wheat, and barley are being used as feedstock for ethanol production. Lately, research on debranning process has been incorporated prior to ethanol fermentation (Wang et al. 2007) to increase the ethanol plant productivity. It is aimed at improving the ethanol quality by increasing the starch percent of the debranned kernels used for ethanol production. Debranning was used successfully by Sosulski and Sosulski (1994) to achieve improved ethanol yield. Moreover, the co-products of ethanol production, such as bran from the debranning process, condensed distiller solubles (CDS), and distillers dried grains (DDG), can be used as animal feed ingredients (Mathews and McConnell 2009; Schroeder 2010).

In general, many grains and their by-products provide carbohydrates and energy to animals when they are incorporated as feed ingredients (Maner 1985). Cereal grains have been used as energy and protein supplement in finishing beef cattle, although forage diets are equally effective (Dinius et al. 1975). In ruminants, wheat and corn provide more than 70% of dry matter (DM) as starch (Huntington 1997). Some of the grain-based raw materials readily available for animal feed production are broken wheat, corn, gram husk, and wheat bran. Among the mentioned, studies have established wheat bran as an animal feed ingredient with high potential. Wheat bran is used as a protein source in many countries (Picard et al. 1993, as cited in Ali et al. 2008). Improved palatability of ruminant diets and a low feeding value were the other benefits observed. These studies support the recommendations of including wheat bran in animal diets. However, additional information is required to establish the significance of feeding wheat bran to animals.

CDS, a co-product of ethanol processing, is obtained by concentrating thin stillage through evaporation and has been used extensively as feedstuff for livestock. When CDS was added to soybean-based diets, animals showed improvement in weight gain (Trenkle 1997). In other instances, researchers observed that a CDS-based diet was effective for lactating cows (Schingoethe 2004). Replacing a part of the corn by CDS in cattle diets improved the performance of cattle (Pesta et al. 2011). Even though some studies have been conducted on CDS, the capacity of CDS as animal feed has not been explored adequately and it needs to be investigated further. The composition of CDS sourced from an ethanol plant in Southern Saskatchewan is given in Table 4.1 (Mosqueda and Tabil 2011).

With the availability of both these feed ingredients (bran and CDS), there is also a requirement for processing them for animal utilization. Processing technologies for improved

animal intake is another issue addressed in this study. Pelleting is recognized as an important part of animal feed manufacture. It improves feed nutrient availability, palatability (Fairfield 2003), weight gain (Briggs et al. 1999), and bulk density. Pelleting causes starch gelatinization by combining moisture and heat on feed ingredients (Kenny and Rollins 2007), thus allowing better utility of nutrients in the feed. The benefits of pelleted feeds, in terms of feeding efficiency, has been reported by Jensen and Becker (1965) in young pigs, Kertz et al. (1981) in dairy cows, and Behnke (1994, as cited in Fairfield 2003) in swine and broilers. Since feed utilization affects animal performance, the physical quality of feed pellets and factors influencing them has been researched. Feed pellet quality (durability and hardness) is affected by several factors, among them - process parameters (Thomas et al. 1997), feed formulation, and steam conditioning (Kenny and Rollins 2007) are the most significant.

Table 4.1 Composition of wheat condensed distillers solubles (Mosqueda and Tabil 2011)

<b>Nutritional component</b>	<b>Composition (% dry matter)</b>
Protein	45.82
Ash	9.30
Fat	2.21
Neutral detergent fiber	18.14
Acid detergent fiber	6.66

The objective of this study was to determine the effect of combining wheat bran and condensed distillers solubles on the physical and nutritional properties of the feed pellets produced. This study was also aimed at optimizing the pelleting process and producing

dimensionally stable feed pellets. A single pelleting study was conducted to optimize the variables for the pilot-scale pelleting study.

### **4.3 Materials and Methods**

#### **4.3.1 Materials**

The experimental materials consisted of wheat grains and CDS. The wheat grains (AC Andrew, a soft white spring variety) were obtained from an ethanol plant located in southern Saskatchewan, Canada, and they were placed in large plastic bins to protect the grains from rodent infestation. The grains were passed through the dockage tester (Opoku et al. 2003a) and the aspirator to separate the grains from the broken kernels, stones, and other grains, if present, prior to debranning (George et al. 2010). The cleaned wheat grains were subjected to debranning in the Satake mill (200 g sample size, 1515 rpm rotational speed, 40 grit size, and 60 s retention time) to accumulate sufficient sample to run the single-pelleting and pilot-scale pellet mill. With optimal bran production rate (12-16% bran fraction per run), almost 10 kg of bran was generated for the pelleting study. The bran was accumulated in Ziploc bags, and placed under specified controlled conditions. Wheat-based CDS was acquired from the same ethanol plant, and frozen in Ziploc bags until it was used. Prior to the experiment, CDS was removed from the freezer and thawed for 4-5 h in the sample preparation area to equilibrate it with the room temperature.

#### **4.3.2 Pelleting equipment**

Single Pelleter: A single pelleter unit with a plunger die assembly (Adapa et al. 2002) was used to study the pelleting characteristics of bran-CDS feed. A cylindrical die, 125 mm and 6.35 mm internal length and diameter, a plunger, and a crosshead were the important components of the

pelleter (Kashaninejad and Tabil 2011). The movable crosshead was connected to the plunger (Instron Model 1011, Instron Corp., Canton, MA; Adapa et al. 2006). The die temperature was regulated, using a heating element. Thermocouples, attached to the temperature controller and the die wall, monitored the pelleting temperature (Tabil 1996). The stainless steel die base had a hole for ejecting the pellets formed. The base supported the pelleter assembly, aiding the vertical movement of the plunger (Kashaninejad and Tabil 2011).

Pilot pellet mill: A pilot-scale pellet mill (Laboratory Model CL-5, California Pellet Mill Co., Crawfordsville, IN) was used to produce feed pellets. The pellet mill had two components: a conditioning chamber and the pelleting assembly. A feed hopper, installed at the top of the pellet mill, controlled the material flow (Tabil 1996). The conditioning chamber was 830 mm in length and 102.7 mm in diameter. A steam supply line supplied superheated steam into the conditioning chamber (Tumurulu et al. 2010). The pellet mill head consisted of a roller with a ridged surface and a rotating ring die assembly (Tabil 1996; Adapa et al. 2004). The feed mixture was conditioned in the conditioning chamber, transmitted to the pelleting chamber, and compressed into the die holes. The roller mill speed was 250 rpm, and it was driven by a 1.5 kW motor (Adapa et al. 2010).

Similar to the single pelleter, thermocouples were used to measure the pre-conditioned mixture temperature, conditioned mixture temperature, and pellet temperature (Tabil 1996). A personal computer connected to the mill acquired the pelleting process data (infeed temperature, conditioning chamber temperature, temperature of mash as it entered the pelleting chamber, pellet temperature as it existed the die hole, and power draw of the mill) recordings (Adapa et al. 2004). In this study, the specific energy consumption was expressed as kWh of energy consumed per tonne of pellets. Throughput rate was measured by weighing the pellets generated.

### **4.3.3 Experimental methods**

The experiments were divided into two stages. The first stage was the single pelleting of bran-CDS samples to optimize the variables affecting pellet quality. The second stage was the pilot-scale pelleting. The results (optimized variables) from the single pelleting studies were validated to produce durable and dimensionally stable pellets.

#### **4.3.3.1 Experimental plan**

The experimental plan for pelleting of feed in the single pelleting unit followed in this study is given in Table 4.2. The combinations of bran-CDS ratio and pelleting temperatures used in the single pelleter unit are also given.

#### **4.3.3.2 Sample preparation**

The bran was mixed with CDS in varying ratios (90% bran:10% CDS, 80% bran:20% CDS, and 70% bran:30% CDS). In order to ensure uniform mixing of the bran and CDS, manual mixing was done. The moisture content of the bran-CDS mixture was determined before the pelleting experiment commenced. At the onset, the bran-CDS mixture was dried to 12-14% moisture content (%wb), based upon literature, to produce high density and durable pellets (Muirhead 1999, as cited in Fairfield 2003).



Table 4.2 Experimental plan of single pelleting unit

Materials	Wheat bran
	Condensed distillers solubles (CDS)
Method of pelleting	Single pelleter
Bran-CDS ratio	70:30
	80:20
	90:10
Pelleting temperature, °C	60
	75
	90

#### 4.3.3.3 Pelleting (making single pellets)

The bran and CDS combination samples were compressed and pelleted in a single pelleting unit under varying temperatures (60, 75, and 90°C), which were also used by Adapa et al. (2002) in pelleting biomass samples. Approximately 0.8-1.0 g of the combined bran and CDS mixture sample was loaded into the cylindrical die. The temperature of cylindrical die was maintained at required levels during pelleting. The Instron machine provided a force of 5000 N to compress the samples (Tabil 1996). The plunger crosshead moved at a speed of 50 mm/min. The plunger compacted the feed material by direct compression. Single-pelleting is a batch process. The relaxation time, after compression, was 60 s. After 60 s, the steel base was removed. Another steel plate with a hole in its center replaced the base for pellet ejection (Tabil 1996). The pellets were ejected from the die using the plunger moving with a speed of 50 mm/min (Kashaninejad et al. 2010). The mass and the dimensions of the pellets were measured.

The pellets were stored in Ziploc bags for 14 d before they were tested for durability and dimensional stability.

#### **4.3.3.4 Pilot-scale pelleting procedure**

The pelleting experiments were performed in CPM CL-5 pellet mill. Steam conditioning (235-250 kPa) was turned on before running the pellet mill. Initially, ground wheat or barley with about 1-2% added oil, was run through the mill to avoid clogging (Tabil 1996). The pellet mill was started when the conditioning chamber reached 95°C (Adapa et al. 2010). The sample material (bran-CDS mixture) was poured into the hopper. As the feed material passed the conditioning chamber and reached the roller-die assembly, they were compacted by continuous layer-by-layer deposition in the die hole. On compression by the roller, pellets were formed. When pelleting started, the data was collected after 180-300 s (Tabil 1996), the time required for system stabilization. The temperature of the pellets was measured by placing a thermocouple in the collection bucket (Adapa 2011). The throughput rate was calculated by weighing the amount of pellets collected. The hot pellets were then cooled and dried to room temperature (Adapa et al. 2010). Similar to single pelleting, the mass, length, and diameter of the pellets were measured. The pellets were stored in Ziploc bags for 14 d before they were tested for durability and dimensional stability.

#### **4.3.3.5 Evaluation methods**

##### Moisture content determination

Moisture content of the bran-CDS samples was reduced to 12-14% by using the heat pump assisted cabinet dryer (35°C and 40% RH) to closely approximate the typical moisture

content needed for feed pellets. Moisture content of bran-CDS samples was determined using a laboratory oven (wet basis). The moisture content was analyzed by drying 2-3 g of the samples (ground) at 135°C for 2 hours (AOAC Method 930.15, AACC 1995). The moisture content was represented as the percentage of the total weight i.e. mass of water/initial weight. For the single pelleting and pilot-scale pelleting unit samples, the moisture content of the bran-CDS samples was analyzed in three replicates.

### Bulk Density

Bulk density of the bran-CDS samples was measured using the bulk density apparatus (SWA951, Superior Scale Co. Ltd., Winnipeg, MB) for grains. The samples were placed on the funnel which sat on top of a 0.5 L steel cylinder (Kashaninejad et al. 2010). Since the samples in the funnel bridged, they were stirred with a wire so that they dropped continuously in to the steel cylinder. The samples were leveled in the cylinder with a steel rod and weighed (Adapa et al. 2009). On dividing the mass of the samples in the steel cylinder by the volume of the cylinder, the bulk density of the samples was calculated (Adapa et al. 2010). Three replicates of the bulk density test were performed for each sample.

### Durability determination

*Durability test for single pelleting:* The durability of the pellets (obtained from single pelleting unit) was measured by conducting the drop-test (Shrivastava et al. 1989; Sah et al. 1980). Single pellets (10 pellets per sample, selected at random from the different bran-CDS pellet combinations) were weighed and dropped from a height of 1.85 m on a metal plate (Tumurulu et al. 2010; Kaliyan and Morey 2009). On droppings, the pellets tend to break due to impact. The

weight retained was noted, and expressed as a percentage of the initial weight of the pellets (Khankari et al. 1989). Each drop test was replicated ten times.

Durability test for pilot-scale pelleting: For the pellets obtained from the pilot-scale pellet mill, the ASAE S269.4 method was used for durability determination (ASAE 1997). Durability of pellets was determined by tumbling 100 g of the sample for 10 min at 50 rpm in a dust-tight enclosure (Adapa et al. 2010). The mass of the remaining pellets was weighed and expressed as percentage of the total mass of pellet sample used during the test. Three replicates of the durability test were performed for each sample.

#### Dimensional stability analysis

The pellets manufactured using the single pelleting unit were collected and stored in Ziploc bags. Ten pellets from each set of samples (varying in bran-CDS ratio) were selected randomly. A digital caliper was used to measure the pellet dimensions (length and diameter) and an electronic balance with a 0.01 mm precision measured the weight of the pellets. The length and diameter values of the pellets were used to calculate the pellet volume. Pellet density was expressed as the mass and volume ratio (Shankar et al. 2007; Shankar et al. 2008). The density values of ten pellets were used to calculate the average pellet density for each bran-CDS combination.

#### Color

Color of the samples was determined using the HunterLab spectrophotometer (Hunter Associates Laboratory Inc., Reston, VA). The whole bran-CDS pellet samples were placed on

top of the colorimeter sensor in a transparent Petri dish (Adapa et al. 2004). The HunterLab color procedures were used to quantitatively assess the lightness (L), redness (a), and yellowness (b) of the bran-CDS samples. The experiment had three replicates.

#### Nutrient composition

Nutritional analysis of the pellets produced was conducted according to standard procedures. Moisture (as loss on drying) in feed materials was determined in triplicates by the Air-oven method: drying at 135°C for 2 h (AOAC 2000a); and the crude protein content in the feed was determined by Kjeldahl Method (AOAC 2000b). Soxhlet extraction method with diethyl ether was used for crude fat determination (AOAC 2000c). The AOCS Ba6a-05 Method (AOAC 1996) was used to determine crude fiber in feeds using the filter bag technique. Gross energy (GE) was determined by measuring the heat of combustion in the samples using a Parr 1281 Bomb calorimeter (Parr Instrument Co., Moline, IL). The laboratory facilities of the Department of Animal and Poultry Science were tapped in conducting these analyses. Starch analysis was carried out by following the procedure as stated in AOAC Method 996.11 (AOAC 1998) and AACC Method 76.13 (AACC 2000) for total starch assay (amyloglucosidase/ $\alpha$ -amylase method).

#### **4.3.3.6 Specific energy calculation for pilot-scale pelleting**

Energy consumption of the CPM CL-5 pellet mill motor was monitored during the pelleting process. It depended on the ingredient formulation, the pelleting process variables, and feed ingredient composition (Tumurulu et al. 2011). Specific energy consumption for processing feed pellets was determined using the methodology of Mani et al (2006a) and Adapa et al.

(2006). The power drawn when the pellet mill was running empty was deducted from the power drawn when the pellet mill was processing the feed pellets. The area under the force-displacement curve, integrated using the trapezoid rule (Cheney and Kincaid 1980, as cited in Adapa et al. 2009) and pellet mass, together yielded the specific energy values in MJ/t.

#### **4.3.3.7 Statistical analysis**

Statistical analysis of the results obtained for pelleting was done using SAS Version 9.2 Software (SAS 2008). The GLM and ANOVA procedures were used in the Student-Newman-Keuls test. Analysis of variance will be used to determine if differences between treatments are significant at 5% significance level. The analysis was useful in determining the experimental variables which have significant effect on the pelleting process.

### **4.4 Results and Discussion**

Experiments were conducted to determine the optimal process conditions in the single pelleting unit and the pilot-scale pellet mill. The wheat bran-CDS mixture was first pelleted in the single-pelleting unit to optimize the pelleting process variables. The compaction characteristics of the bran-CDS mixture were studied using the single-pelleting experiments. The single-pelleter and pilot-scale mill varied in the method of compaction. The former is a batch process whereas the latter is a continuous process. Single-pelleting test was done to screen the samples based on their pelleting quality. The results of the single-pelleting study could be used in the pilot-scale mill to develop high-quality pellets. Therefore, the pilot-scale pelleting was done to verify the optimized variables from the single pelleting studies, and to produce feed pellets having good physical and nutritional quality

#### **4.4.1 Optimizing the variables in the single pelleting unit**

The experiments in the single pellet mill involved varying the ingredient formulation and parameters of the single pelleting unit to produce dimensionally stable and durable pellets.

##### **4.4.1.1 Moisture content**

The moisture content of the bran-CDS samples was varied in order to convert the mixed mash to a physical state which was more suitable for pelleting. In the results obtained, the moisture content of the mixture measured before pelleting the mash, after pelleting, and after 14 d of storage showed a regular pattern of decrease with increase in the number of days after pelleting. The results confirmed that the temperature used for pelleting (60, 75, and 90°C) resulted in reduction in the pellet moisture content. This implied that the moisture content of the bran-CDS mash was higher than the moisture content of the pellets. The moisture content of the bran-CDS mixture (90% bran:10% CDS, 80% bran:20% CDS, and 70% bran:30% CDS) and pellets manufactured under varying pelleting temperatures are shown in Table 4.3.

An optimal moisture level of feed mixture improves process economics by decreasing the energy consumption during pelleting. In addition, it prevents any hindrances in pellet manufacturing (van der Heijden and de Haan 2010). A steady production prevents excessive heat generation, which in turn, reduces nutrient losses. Furthermore, pellets having good properties are formed at an optimal moisture level, such as pellets with good durability. Mani et al. (2006b) stated that the density of biomass pellets and the moisture content were inversely proportional.

Statistical analysis indicated that moisture content played a significant role on pellet durability, dimensional stability, and density. Variation in the moisture content of the mixture also leads to property changes in the components, such as starch and protein. The presence of moisture brings the adhesive properties of the pellets on the surface and hence, improves the pellet quality. The presence of water is a prerequisite for gelatinization. On cooling, the gelatinized starch cools and forms a gel, functioning as an adhesive for the particles (Lund 1984, as cited in Moritz et al. 2005). Similar to changes in starches, the protein component of the bran-CDS samples undergo denaturation, forming an insoluble protein gel, improving the adhesive properties of the pellets (Wood 1993). In this study, the moisture content available during pelleting may have caused partial starch gelatinization and protein denaturation. However, partial starch gelatinization and protein denaturation may not be the only factors affecting pellet quality. Pellet quality was affected by the interaction of all feed ingredients (Buchanan 2008). Hermansson (1979) showed that even incomplete changes in functional components like proteins brought about adhesiveness, improving pellet quality. Statistical analysis confirmed that neither bran-CDS ratio nor pelleting temperature had significant effect on the moisture content of the pellets ( $P = 0.37$  and  $P = 0.77$  respectively, Table B.1). This shows that although pelleting temperature and ingredient formulation affected the feed pellet moisture content, the magnitude of the effect is minimal.



Table 4.3 Moisture content (% wet basis), density, and durability of wheat bran-condensed distillers solubles (CDS) mixture before and after pelleting in the single pelleting unit

<b>Bran-CDS Ratio (%)</b>	<b>MC before pelleting (%)</b>	<b>Pelleting temp. (°C)</b>	<b>MC (%) (Day 1)</b>	<b>MC* (%) (Day 14)</b>	<b>Pellet density** (Day 1, kg/m³)</b>	<b>Pellet density (Day 14, kg/m³)</b>	<b>Pellet durability*** (Day 14, %)*</b>
70:30	14.00	60	9.27(0.04)a	8.35(0.04)a	1221.7(6.5)a	1273.1(16.8)a	96.4(3.8)a
70:30	14.07	75	10.55(0.03)a	9.07(0.04)b	1221.8(20.4)a	1281.9(46.9)a	98.1(1.2)a
70:30	14.43	90	9.51(0.01)a	8.42(0.01)a	1227.8( 9.1)a	1309.4(32.5)a	96.0(4.7)a
80:20	15.00	60	9.83(0.38)a	8.41(0.04)a	1214.3(20.0)a	1254.6(21.1)a	98.2(2.4)a
80:20	14.93	75	9.46(0.04)a	8.23(0.09)a	1225.8(16.4)a	1259.8(10.2)a	97.9(2.6)a
80:20	15.42	90	9.39(0.10)a	8.41(0.12)a	1250.4(25.5)a	1308.2(46.9)a	98.1(2.5)a
90:10	14.50	60	9.81(0.01)a	8.50(0.01)a	1217.1 (11.6)a	1247.7(16.6)a	99.9(0.1)a
90:10	14.93	75	10.42(0.09)a	8.57(0.04)b	1247.6(9.3) a	1281.4(10.4)a	99.2(1.9)a
90:10	15.00	90	10.45(0.07)a	8.64(0.01)c	1267.2(13.3)a	1304.2(17.4)a	99.9(0.2)a

Number in parenthesis is the standard deviation,\*n = 3 replicates, \*\* and \*\*\* n = 10 replicates, a, b, c = indicate that means with the same letter in a column are not significantly different at P = 0.05

#### **4.4.1.2 Pellet density**

In general, the pellet density of the three proportions of bran-CDS samples changed with the pelleting temperature. According to Mani et al. (2003), the temperature and pressure influenced the pellet density. The results obtained in this study showed that pellet density increased with increase in pelleting temperature (Table 4.3). On day 1, the pellet densities for the 70:30, 80:20, and 90:10 bran-CDS ratio were the highest for 90°C pelleting temperature. They were  $1227.8 \pm 9.1 \text{ kg/m}^3$ ,  $1250.4 \pm 25.5 \text{ kg/m}^3$ , and  $1267.2 \pm 13.3 \text{ kg/m}^3$ . After cooling (Day 14), the pellet density increased due to a decrease in the length and the diameter of the pellets. The weight of the pellet decreased slightly on cooling due to moisture loss. On day 14, the pellet densities for the 70:30, 80:20, and 90:10 bran-CDS ratio were highest for 90°C pelleting temperature. They were  $1309.4 \pm 32.5 \text{ kg/m}^3$ ,  $1308.2 \pm 46.9 \text{ kg/m}^3$ , and  $1304.2 \pm 17.4 \text{ kg/m}^3$  respectively. Statistical analysis results showed that bran-CDS ratio and pelleting temperature had positive significant effect on the changes in the pellet density between day 1 and 14 ( $P < 0.01$  for both, Table B.2).

#### **4.4.1.3 Pellet durability**

Pellet durability was determined using the drop-test 14 d after pelleting. Table 4.3 shows the results for durability of pellets. For any bran-CDS ratio, the pellet durability did not show significant change with change in pelleting temperature. The highest pellet durability was attained for 90:10 bran-CDS ratio under all three pelleting temperatures i.e. 60, 75, and 90°C ( $99.9 \pm 0.1$ ,  $99.2 \pm 1.9$ , and  $99.9 \pm 0.1\%$  respectively). This was similar to the results reported by Hill and Pulkinen (1988, as cited in Adapa et al. 2002), where the high pellet temperature (60-104°C) positively influenced pellet durability. This could primarily be due to the relationship

between pelleting temperature and changes in the ingredient characteristics. Statistical analysis showed that only bran-CDS ratio had positive significant effect on the durability of the pellets ( $P < 0.01$ , Table B.3).

According to Thomas et al. (1998), the protein and starch components determine the physical quality of the feed pellets. Studies have revealed that the changes in the functional components positively affected the durability of the pellets (Briggs et al. 1999; Wood 1987). Starch gelatinization at higher temperatures increased particles binding and improved the durability. A high durability of the pellets indicates that the pellets produced would be able to resist breakage during handling prior to consumption (Adapa et al. 2009). High durability is an indicator of good physical quality and mechanical properties (bulk density, pellet density) of feed pellets.

The results for the single pelleting unit indicated that bran-CDS ratio and pelleting temperatures affected the physical properties of the feed pellets. Under varying pelleting temperatures, a variation in pellet density and durability values was observed. On day 1, the highest pellet density for the bran-CDS pellets at 60°C were exhibited by 70:30 ratio, whereas 90:10 ratio exhibited highest pellet density at 75 and 90°C. At 60°C, the pellet density for 70:30 ratio was  $1221.7 \pm 6.5 \text{ kg/m}^3$ , and the pellet densities for 90:10 ratio at 75 and 90°C were  $1247.6 \pm 9.3$  and  $1267.2 \pm 13.3 \text{ kg/m}^3$ . After cooling (Day 14), the pellet density of 70:30 ratio increased more than 80:20 and 90:10 ratio. For all pelleting temperatures, the 70:30 bran-CDS pellets exhibited the highest pellet densities. With regard to durability, the 90:10 bran-CDS pellets exhibited the highest values for all pelleting temperatures. The single pelleting study concluded that the optimal conditions for production of feed pellets were high pelleting

temperatures for all bran-CDS ratios. Overall, 90°C pelleting temperatures displayed good physical characteristics, although the physical properties for other pelleting temperatures were not significantly different. Similarly, 90:10 ratio pellets displayed good physical characteristics, although the physical properties for the other bran-CDS ratio pellets were not significantly different. The optimal conditions during the single pelleting test were tested and verified further in the pilot-scale pellet mill.

#### **4.4.2 Pilot-scale pelleting**

The quality of pellets may be affected by process variables, such as dimensions of the die (Tabil and Sokhansanj 1996) and the speed of the rollers in the mill (Adapa et al. 2004). The experiments in this stage involved varying the ingredient formulation only to produce dimensionally stable and durable pellets. At the onset of the pelleting experiments, the ground and uniformly mixed bran-CDS samples (70:30, 80:20, and 90:10 ratio, 2 kg each) were dried to 12-14% moisture content (% wb). The steam conditioner of the pilot-scale pellet mill served to add moisture and heat to the bran-CDS mixture during processing. The pellets produced from 70:30, 80:20, and 90:10 bran-CDS ratio had an average length of  $20.49 \pm 0.85$ ,  $19.76 \pm 0.80$ , and  $18.73 \pm 1.25$  mm, respectively on day 1, and  $20.27 \pm 0.81$ ,  $19.49 \pm 0.72$ , and  $18.45 \pm 1.27$  mm, respectively on day 14. Improvement in particle-bonding during storage reduced the pellet length. The pellet length of all the three bran-CDS pellets in this study showed a consistent reduction.

#### 4.4.2.1 Moisture content

Moisture content affects the machine operation and product quality during feed manufacturing. Studies by Muirhead (1999, as cited in Fairfield 2003) have revealed that precise moisture control has a positive effect on feed pellet durability. The study concluded that the initial moisture content for improved pellet production was 13-14%. For this reason, the initial moisture content of the bran-CDS mash in this study was maintained at 12-14%. The results for the moisture content of the mixture measured before pelleting, after pelleting, and after 14 d of storage are shown in Table 4.4. After processing and on cooling, a decrease in the moisture content of the feed material was observed, indicating that the different process variables lead to moisture loss from the feed pellets.

Table 4.4 Moisture content (% wet basis) of bran-CDS mixture before and after pelleting in the pilot-scale pellet mill

<b>Bran- CDS ratio</b>	<b>Moisture content before pelleting (%)</b>	<b>Moisture content (Day 1, %)</b>	<b>Moisture content change (before and after pelleting, %)</b>	<b>Moisture content (Day 14, %)</b>	<b>Moisture content change (Day 1 and 14, %)</b>
70:30	13.61(0.02)ab	12.06(0.28)a	11.41	11.60(0.24)a	3.75
80:20	13.77(0.10)a	10.28(0.13)b	25.38	9.98(0.07)b	2.91
90:10	13.47(0.13)b	9.81(0.13)c	27.19	9.38(0.24)c	4.39

Number in parenthesis is standard deviation; a, b, c = indicate that means with the same letter in a column are not significantly different at P=0.05

#### 4.4.2.2 Bulk density

The bulk density values for bran-CDS samples before and after pelleting, and on the Day 14 are given in Table 4.5. The bulk density of feed materials affects the handling, transporting, and storage of the feed. The bulk density of wheat bran was 317 kg/m<sup>3</sup>. When wheat bran was added to CDS in three proportions and pelleted, the bulk density of the mix improved.

Table 4.5 Bulk density, pellet density, and pellet durability before and after pelleting, and throughput rate and specific energy consumption during pelleting of bran-CDS mixture in the pilot-scale pellet mill

Parameters	Bran-CDS ratio		
	70:30	80:20	90:10
Bulk density of mash (before pelleting, kg/m <sup>3</sup> )	496.4(1.0)a	502.6(0.5)b	375.5(2.0)c
Bulk density of pellets (Day 1, kg/m <sup>3</sup> )	751.4(3.5)a	764.3(2.6)b	765.5(3.1)b
Bulk density of pellets (Day 14, kg/m <sup>3</sup> )	760.9(2.0)a	770.2(2.7)b	774.5(2.1)b
Pellet density (Day 1, kg/m <sup>3</sup> )	1210.4(42.8)a	1210.1(67.6)a	1244.8 (44.9)a
Pellet density (Day 14, kg/m <sup>3</sup> )	1219.1(51.3)a	1243.6(44.2)a	1245.7(23.6)a
Pellet durability (Day 1, %)	97.4(0.3)a	96.7(0.5)a	94.0(0.9)b
Pellet durability (Day 14, %)	97.8(0.8)a	97.0(0.1)a	94.1(0.1)b
Throughput rate (kg/h)	23.61	25.27	26.30
Specific energy consumption (MJ/t)	88.7	82.3	79.2

Number in parenthesis is standard deviation; a, b, c = indicate that means with the same letter in a row are not significantly different at P = 0.05

The highest bulk density was 502.6 kg/m<sup>3</sup>, obtained for 80:20 bran-CDS ratio. On pelleting, the bulk density increased by about 1.5 times for all three bran-CDS mixtures. The highest change was observed in 90:10 ratio (51.0%). The improvement in bulk density is

attributed to densification of feed particles and other factors. Larsson et al. (2008) demonstrated that the moisture content had the most significant effect on pellet bulk density. They found that increasing the moisture content lead to a decrease in bulk density. Similar observations were made by Fasina (2008) in experiments based on peanut hull pellets. The results of this test agreed with these findings. The highest bulk density was observed for 90:10 bran-CDS mixture, having lowest moisture content of  $9.81 \pm 0.13\%$ . The results for statistical analysis are given in Table B.4. Statistical analysis showed that bran-CDS ratio, moisture content, and retention time had positively significant effect ( $P < 0.01$  for each factor) on bulk density. Also, the interaction between ratio and moisture content affected bulk density positively and significantly ( $P = 0.03$ ).

#### **4.4.2.3 Pellet durability**

Durability is the force-bearing capacity of pellets during transport, and it is the most important physical quality of a pellet (Tabil and Sokhansanj 1996). Durable pellets contribute in preventing the dissociation of ingredients (Buchanan and Moritz 2009), improving the palatability (Winowski 1995) and the intake of the feeds. High durability value is greater than 80%, medium is between 70-80%, and low is less than 70% (Colley et al. 2006, as cited in Theerarattananon et al. 2011). According to studies conducted by Kenny and Rollins (2007), with variation in diet formulations and the conditioning action, the pelleting durability varies.

In general, all the bran-CDS ratios formed highly durable pellets (Table 4.5). The lowest pellet durability was seen in 90:10 bran-CDS pellets ( $94.0 \pm 0.9\%$ ) and the highest durability was seen in 70:30 ratio pellets ( $97.4 \pm 0.3\%$ ). On cooling and storage (Day 14), the durability of the pellets further increased. Though pellet durability did not increase significantly on cooling, the reason for the increment could be recrystallization of solubilized sugars. The recrystallized

sugars formed bonds between feed particles and strengthened the feed pellets (Thomas et al 1998). The highest durability was seen in 70:30 ratio pellets ( $97.8 \pm 0.8\%$ ) which showed the maximum change in pellet durability upon cooling. Durability of pellets was negatively correlated to the pellet mill throughput, and was positively correlated to the specific energy consumption (Table 4.5). Statistical analysis are given in Table B.5 which showed that only bran-CDS ratio had positively significant effect ( $P = 0.01$ ) on pellet durability.

In the experiments conducted, 17 to 20 s of residence time (Tabil 1996) in the steam conditioning chamber, and 13-14% moisture content (initial) of the bran-CDS mixture were the conditions maintained. Higher retention time increases the degree of gelatinization (Kenny and Rollins 2007), thereby improving the durability of the pellets. Slower rotation in the conditioner (Gilpin et al. 2002) and varying the paddle pitch (Briggs et al. 1999) of the steam conditioner increase the retention time of the feed mash.

#### **4.4.2.4 Dimensional stability of pellets**

The dimensional stability of the pellets was calculated by measuring the dimensions of the pellets after pelleting and on cooling and storage are shown in Table 4.5. On day 1, the highest density was  $1244.8 \pm 44.9 \text{ kg/m}^3$  for 90:10 bran-CDS pellets and lowest was  $1210.1 \pm 67.6 \text{ kg/m}^3$  for 80:20 bran-CDS pellets. Pellet density changed tremendously on cooling, which implied that the bond between the feed particles strengthened. The change in pellet density was the highest in the case of 80:20 ratio pellets (2.7%).



#### **4.4.2.5 Color**

The results for color analysis of the bran-CDS mixture and bran-CDS pellets at day 1 and 14 are given in Table 4.6. The Hunterlab L values ranges from 0 (black) to 100 (white). The Hunterlab a and b values imply redness and yellowness respectively (Cromwell et al. 1993). Also, L values greater than 60, as in the case of the bran-CDS mash, imply that the samples were light in color (Table 4.6). The Hunterlab L values for pelleted samples ranged from 55.50 (80:20 bran-CDS ratio after pelleting) to 47.36 (70:30 bran-CDS ratio after pelleting). According to the Hunterlab L values, the three bran-CDS samples fell into two main categories: the three bran-CDS mash (lightest) and the three bran-CDS pellet samples on day 1 and 14 (medium). The results indicate that browning could have taken place during pelleting due to heated conditions in the pellet mill, leading to darkening of the feed pellets. The Hunterlab b (yellowness) values ranked the three samples in the same order as the L values, but the Hunterlab a (redness) values did not.

Table 4.6 Color and nutritional characteristics of bran-condensed distillers soluble (CDS) pellets from the pilot-scale pellet mill

Parameter			Bran-CDS ratio		
			70:30	80:20	90:10
Color parameters	Before pelleting (mash)	L	61.00	63.88	66.68
		a	5.03	3.74	3.90
		b	16.41	14.30	14.19
	Day 1 (whole pellets)	L	47.36(0.83)a	55.50(0.97)b	50.70(0.31)c
		a	4.00(0.20)a	4.28(0.26)a	4.13(0.49)a
		b	11.77(0.24)a	13.57(0.98)a	12.42(1.04)a
	Day 14 (whole pellets)	L	49.65(0.61)a	50.24(1.41)a	49.93(1.34)a
		a	4.20(0.29)a	4.13(0.12)a	3.67(0.11)b
		b	12.60(0.46)a	12.24(0.36)a	11.22(0.47)b
Nutritional composition (% DM basis)	Dry matter (%)		87.94	89.72	90.19
	Crude protein (%)		18.42	17.57	16.57
	Fat (%)		4.38	4.00	3.93
	Crude fiber (%)		63.80	61.37	61.06
	Starch (%)		9.05	9.08	9.81
	Gross energy (MJ/kg)		18.93	18.60	18.44

Number in parenthesis is the standard deviation; a, b, c = indicate that means with the same letter in a row are not significantly different at P = 0.05; L, a, b = lightness, yellowness, and redness, N = 3 for pellet density and L,a,b, and N = 2 for nutritional composition

#### **4.4.2.6 Nutritional composition**

Prior to pelleting, the bran and CDS were analyzed individually for their nutritional composition. Wheat bran had 16.16% crude protein, 4.14% fat, 8.04% crude fiber, 17.39% starch, and 4529.10 MJ/kg of gross energy. The CDS had 45.82% crude protein, 2.21% fat, 18.14% NDF, 6.66% ADF, and 9.30% ash. CDS contains substantially more protein, fiber, and fat than the whole wheat grain. The high protein value of CDS makes it a protein supplement in animal feeds. In general, the bran-CDS combination demonstrated a high protein, fat, and crude fiber content. Table 4.6 enumerates the average chemical composition of the three wheat bran-CDS pellet samples for tests performed in duplicates. The 70:30 ratio had the highest protein (18.42%), fat content (4.38%), gross energy value (18.93 MJ/kg), and fiber content (63.80%); the 90:10 ratio had the highest starch (9.81%) and dry matter content (90.19%). Thus, the bran-CDS pellets can be considered as feed ingredient for poultry, ruminants, and swine.

#### **4.4.2.7 Specific energy consumption**

The energy requirements of the pelleting process are crucial in its economic viability (Adapa et al. 2009). The specific energy consumption values for the pilot-scale pellet mill are given in Table 4.5. The energy requirements of the pellet mill for 70:30, 80:20, and 90:10 bran-CDS ratios were 88.7, 82.3, and 79.2 MJ/t respectively. The specific energy consumption values obtained were quite high as compared to the values obtained by Thomas et al. (1998) for cotton seed meal (23.4MJ/t) and soy-bean meal (20.2 MJ/t). The values obtained ranged between 151.8-228.7 MJ/t. In this study, the specific energy requirements and the throughput rate were inversely proportional (Table 4.5). Similar observations were made by Tumurulu et al. (2011) who stated

that specific energy and throughput rate were negatively correlated. The pelleting conditions and feed ingredients used affected the energy consumption, as reported by Thomas et al. (1998).

#### **4.5 Summary and Conclusion**

The experiments using a single pelleting unit indicated that the bran-CDS ratio and pelleting temperatures affected the physical characteristics of feed pellets. The experiments concluded that the optimal temperatures for production of feed pellets in the single pelleting unit are higher for all bran-CDS ratios. Specifically, the 90:10 bran-CDS ratio, at 90°C pelleting temperature, produced feed pellets of higher durability ( $99.9\pm0.2\%$ ) and pellet density ( $1304.2\pm17.4\text{ kg/m}^3$ ), but the physical quality values were not statistically different from the other bran-CDS ratios.

The pilot-scale pelleting results showed that bran-CDS ratio, moisture content of the samples, and pelleting conditions affected the physical and nutritional characteristics of the feed pellets. In general, 70:30 and 90:10 ratio pellets had extreme values for most factors. The 70:30 ratio had a bulk density of  $751.4\text{ kg/m}^3$ ,  $97.4\pm0.3\%$  durability,  $23.61\text{ kg/h}$  throughput rate,  $88.7\text{ MJ/t}$  specific energy, and  $210.3\pm42.8\text{ kg/m}^3$  pellet density. Upon cooling, the bulk density, durability, and pellet density changed. The change in bulk density and durability was observed to be the highest for 70:30 ratio, indicating an improvement in the physical characteristics. With significantly higher durability values, the 70:30 bran-CDS ratio pellets exhibited physical characteristics desirable for handling and transportation. A better nutritional profile of 70:30 bran-CDS ratio (18.42% crude protein, 4.38% fat, 63.80% fiber, 9.05% starch, and 18.93 MJ/kg gross energy) makes it the choice for production of feed pellets having good physico-chemical characteristics.

## **CHAPTER FIVE**

### **GENERAL DISCUSSION**

This thesis explored the prospects of processing wheat bran and condensed distillers solubles (CDS), the co-products of ethanol production, as animal feed. It covered the processing techniques which can be utilized for optimal production of feed pellets. The thesis also aimed at finding ways to address one of the perennial problems the bioethanol industry faces when it comes to utilization of the substantial amount of available co-products. Studying and controlling the process variables involved in wheat bran production and the pelleting process provides a potential scope for developing high quality (in terms of density and durability) feed pellets. The study was divided into two parts: firstly, to optimize the debranning process, aimed at maximizing the bran production; and secondly, analyzing the pelleting characteristics of bran-CDS mixtures and developing them into feed pellets having good physical and nutritional characteristics.

Although debranning of wheat using abrasive equipment has been done before, it has not been incorporated in wheat-based ethanol production using dry milling process. Optimizing the process and selecting the abrasive equipment with higher debranning efficiency was essential to improve the starch content of the debranned kernels, increasing the ethanol yield. Not only does abrasive dehulling improve the yield of dehulled grains, it also permits controlled removal of bran layers (Reichert and Youngs 1976), and improves the rate of hull removal. Studies have shown that the reproducibility of abrasive dehullers was very good (Oomah et al. 1981). In this study, it was found that the debranning process can be improved by optimizing the sample size, retention time, grit size, and rotational speed. It was also determined that, on a laboratory-scale,

the Satake mill was more suitable for the process as compared to the TADD mill. In the Satake mill, process variables such as retention time and rotational speed had a significant effect on bran yield. Higher retention time and rotational speed were positively correlated to bran production. Increasing the retention time (60-90 s) would increase the residence time of wheat grains in the mill, thereby lengthening the duration of abrasion. Reichert et al. (1984), working on the TADD mill, had concluded that between 60 and 180 s dehulling time was linearly correlated to the bran produced. Wang (2005) obtained contradicting results in case of the Satake mill, and had concluded that at a higher speed, decreasing the retention time ( $< 40$  s) increased the dehulling efficiency. Furthermore, higher rotational speed caused more mechanical friction between the grain kernels and the abrasive wheel, resulting in enhanced bran production. These results contradicted with the findings of Reichert et al. (1979) who stated that higher speeds reduced dehulling efficiency in the TADD mill. With variance in sample size, the pearling index showed significant differences. Moreover, sample size of the grains was associated with improved bran production. McCluggage (1943, as cited in Lawton and Faubion 1989), when investigating factors affecting wheat kernel hardness in the TADD mill, reported that pearling index was affected by sample size. A larger sample size (200 g) lowers production costs, making the process more economical. In this study, besides sufficient bran yield, using the Satake mill resulted in a good starch separation from the wheat grains, thereby rendering them as feedstock for ethanol production. The study by Sosulski et al. (1997) drew similar conclusions. A higher residence time in the dehulling mills indicated that more dehulling units needed to be employed for dehulling in the commercial level. This implies that the investment in capital and operational costs, and the power requirement for running the dehulling units would be higher.

Starchy grains, like corn and wheat, have been used as feedstock for ethanol production (Turhollow and Heady 1986). Debranning, as mentioned earlier, has been incorporated as a preprocessing technology in manufacturing ethanol. As a result, the starch content of the dehulled grains increased. Studies by Sosulski and Sosulski (1994) showed that debranned kernels were suitable for subsequent milling and fermentation. With almost 10-11% increase in the starch content in the debranned flour, the fermentation capacity of the ethanol plant increased by 20.3-26.4%, the ethanol concentration increased by 11.9% in the beer, and the ethanol production capacity of the plants increased by 8-23% (Sosulski et al. 1997). Consequently, the value of the co-products improved, and the process was commercially beneficial (Sosulski and Sosulski 1994). Debranning is also responsible in altering the chemical composition of distillers dried grains with solubles (DDGS), a major co-product from ethanol production (Corredor et al. 2006). An increase in the protein, crude fiber, lipid, and ash content of DDGS was observed by Rasco et al. (1987). To maximize the profitability of fuel ethanol, marketing the co-products was critical. Two of the co-products, the bran from the debranning process and condensed distillers solubles (CDS), have been incorporated into feed rations.

Wheat bran has been the source of starch and sugars for ruminants and is important for healthy growth of ruminants. It improves digestibility and provides energy for metabolic activities to animals. CDS, on the other hand, is a well-known cattle feed supplement. With high fat and protein levels, CDS provides high amount of energy to animals (Schroeder 2010). In its liquid state, it is used as a water supplement in dry feeds (Lardy 2007). With optimal quantity of bran and CDS available as feed ingredients, animal feed production was the second objective of this study. Methodologies to overcome the barriers in economic use of animal feeds were considered. Processing of animal feed into dimensionally stable and durable pellets is an

effective solution to the transportation and storage problems associated with conventional feed mash (Karkania et al. 2012). In addition, high density is often associated with high pellet durability. At the onset of pelleting, the bran-CDS mixture was dried to 12-14% moisture content. Adjusting the initial moisture content to 12-14% ensured that the feed mash did not have excessive friction and there was sufficient lubrication during pelleting. In addition, the moisture content was proportional to the pellet durability, as reported by Lehtikangas (2001). Uniform mixing of the bran and CDS was important in preventing lump formation, which could be a liability during pelleting. Pelleting studies were done in two stages: a) lab-scale studies were done to optimize the process variables; and b) pilot-scale studies were done to verify the optimized variables. The density of the pellets was observed to significantly increase with an increase in pelleting temperature in the lab-scale single pelleting experiments. Additionally, the bran-CDS ratio had significant effect on pellet durability. Higher physical quality of pellets at high pelleting temperatures could probably be attributed to the gelatinization of starch and denaturation of protein. According to Wood (1987), the changes in the starch and protein components affected the pellet durability more than other factors. In general, pellet durability was the highest for 90:10 bran-CDS ratio under all pelleting temperatures in the single pelleting experiments. Even though single pelleting experiments were adequate in selecting the optimal pelleting parameters, the durability values did not make a clear demarcation among the bran-CDS pellets. This could be due to the test method used. The drop test determines the pellet's ability to survive an impact. Softer pellets, in their fragile state might remain intact when dropped and give high durability values. When softer pellets are transported to long distances, the probability of breakage is higher. Softer pellets are also prone to damage, decreasing pellet



quality by formation of fines. Therefore, the drop test would be inadequate in differentiating between pellets having good physical properties (i.e., hardness, durability).

Pilot-scale pelleting of the bran-CDS mixture was performed in order to determine the feasibility of effectively pelleting the animal feed and to build on the results and conclusions derived from lab-scale pelleting. Among the variables in this study namely, pellet retention time, bran-CDS ratio, and moisture content of the pellets, only the bran-CDS ratio affected pellet durability significantly. Thomas et al. (1998) and Fairfield (2003) stated that nutrient functionality affected the physical properties of pellets. Interestingly, more than ingredient formulation, longer retention times and higher moisture content caused starch gelatinization and solubilization, improving the durability of the pellets. Longer retention times for pelleting increased the time of interaction between the feed particle and the processing conditions (i.e. heat and moisture). The above mentioned processing conditions improved the pellet quality (Fairfield 2003). Gilpin et al. (2002) also suggested that retention time essentially affected energy consumption during the pelleting process.

Moisture content of the pellets played a vital role in the final product quality. Muirhead (1999, as cited in Fairfield 2003) claimed that controlled moisture content influenced pellet durability positively. Tumurulu et al. (2010) had stated that steam conditioning prior to pelleting, increased the physical quality of the pellets formed. Greer and Fairchild (1999) also stated that mash moisture content influenced pellet durability. This study reported that mash moisture content and durability could be correlated. Furthermore, durability of pellets was negatively correlated to pellet mill throughput and was positively correlated to specific energy consumption (Table 4.5). In contrast to the lab-scale results, pellet durability was the highest for 70:30 ratio in pilot-scale pelleting. The tumbling test was used for durability determination. In the tumbling

test, two forces act upon the pellets: particle/particle impact and particle/container impact. The conditions faced by the pellets during the tumbling test were similar to those faced during the transportation and handling process, hence the durability results from the tumbling test are more reliable.

Pilot-scale pelleting was also done to solve another set of practical concerns, primarily lower bulk density and poor nutritional quality of animal feeds. Upon pelleting, the bulk density increased at least 1.5 times the initial value of the feed mash, due to feed particle re-arrangement and compaction. Bulk density was affected by the retention time and bran-CDS ratio significantly. However, the moisture content of pellets and the bulk density were found to be negatively correlated.

In addition to the physical properties, the nutritional quality of the feed improved upon pelleting (Skoch et al. 1983). The nutritional quality was affected by two factors: the nutrient digestibility and the composition of the feeds. Nutrient digestibility of the feeds increases the nutrient accessibility for growth and production. Chae et al. (1997) reported that pelleting of growing-finishing pig diets improved the nutritional digestibility. Similarly, Jensen and Becker (1965) stated that pelleting improved the energy availability for young pigs. On pelleting, the heat generated leads to starch gelatinization, causing the starch molecules to break down, making the feeds more digestible (Ferket et al. 2002) and improving the nutrient utilization (Moritz et al. 2005). The crystalline nature of the starch molecule is destroyed upon gelatinization, making it digestible (Huntington 1997) by rumen enzymes and microbes. Pelleting also leads to protein denaturation (Briggs et al. 1999). According to Svihus and Zimonja (2011), denaturation inactivates the protein antinutrients, thereby improving nutrient absorption. Also, feed conversion efficiency of the animals is improved (Briggs et al. 1999) as a result of pelleting due

to lesser spillage by animals on consumption (Ferket et al. 2002). Thus, pelleting improves the nutrient utilization and digestibility (Fairfield 2003). Uniform nutrient availability is also attained as a consequence of pelleting.

Although the nutritional benefits provided by the bran-CDS pellets can only be measured through animal feeding trials, the nutritional analysis of the pellets showed that the protein, fiber, fat, and starch contents were in desirable levels, making them suitable for animal consumption. The nutritional and the physical quality of the feed pellets were correlated, as the nutritional quality of the pellets is influenced by the process parameters (Skoch et al. 1983), which in turn, influence the physical properties of the pellets. Aside from improving the physical quality of the pellets, steam conditioning also influences the nutrient composition of the feeds (Winowski 1995). The composition of the bran-CDS pellets, in accordance with the animal requirements, is discussed below.

On combining the bran and CDS to produce feed pellets, the fiber and fat content of the pellets improve substantially. In terms of the nutritional composition, the bran-CDS pellets showed high protein and gross energy levels, indicating a good feeding value as an alternative feed ingredient for poultry, ruminants, and swine. It was observed that the bran-CDS pellets have lower protein content compared to CDS, which can be compensated by adding other ingredients to the animal feed. The protein content in animal feeds is an important component. According to Olomu and Offiong (1980), high protein content enhanced the performance of broiler chicks. Parish and Rhinehart (2008) also stated that protein is an important component of beef cattle diets. They concluded that a feed with at least 15% protein was essential for healthy growth of nursing calves. For lactating swine, the protein requirement is 12-20% (Mahan and Grifo 1975). With 16.57-18.42% crude protein content, the bran-CDS pellets could be used as a feed

ingredient for beef cattle and swine. The poultry diet protein content requirement also falls in this range. Fiber is another nutrient of great importance to beef cattle and pigs, improving nutrient digestibility. It maintains the rumen pH (Allen 1997), preventing metabolic disorders. A 61.06-63.80% fiber content of bran-CDS pellets could help ruminants in maintaining digestive functions, but was higher than the requirement of poultry diets. Also, with high gross energy levels, bran-CDS pellets could be used as alternative feeds for ruminants.

With respect to pellet storage/cooling, it was observed that for both the lab-scale and pilot-scale pelleting, cooling of pellets improved pellet quality. Pellet density increased due to enhanced bonding among feed particles. Moreover, recrystallization of sugars may have occurred during cooling and storage, leading to formation of stronger bonds, thus improving pellet durability (Thomas et al. 1998). Equally important, the decrease in the moisture content on cooling (Tables 4.3 and 4.4) reduces the risk and speed of mould growth. Jones (2008) stated that moisture loss on cooling was a quality control factor in pelleted feed manufacturing. On a positive note, pelleting of bran-CDS mixture produced pellets having good physical properties and nutritional value. Thus, developing feed pellets from ethanol production co-products is highly feasible in terms of processing and nutritional aspects.

## **CHAPTER SIX**

### **SUMMARY AND CONCLUSION**

#### **6.1 Summary**

Grain-based convention mixtures are being used as animal feed leading to inflation in animal feed costs. Alternative feeds are required to alleviate this problem. Development of animal feeds which can act as substitute to the ones currently in the market is a requirement. The animal feed should be able to maintain the standards in terms of feed nutritional characteristics, productivity, and cost. Knowledge of the processes needed in the production of animal feed from bran and CDS is critical in controlling the manufacturing process to achieve high production efficiency. Optimal product quality can be ensured by understanding each step involved in the process.

This thesis work addressed two problems. The first problem was to find ways for optimal production of bran to be used as a component of animal feed. Optimal debranning maintained the quality of nutrients in bran and increased the conversion efficiency of grains to ethanol. Experiments were conducted to achieve the maximum bran production from two types of laboratory debranning equipment (Satake and TADD mills) and to determine the optimal debranning conditions. The process variables affecting debranning efficiency were as follows: sample size, rotational speed, grit size, and retention time. The starch separation efficiency of the two mills was also examined to determine the ideal debranning conditions.

The second problem was the production of dimensionally stable feed pellets with good physico-chemical characteristics. To address this issue, experiments were undertaken to study the pelleting characteristics of the bran and CDS as animal feed. Pelleting studies were done in

the single pelleting unit and the pilot-scale pelleting mill to develop pellets with higher attributes. The process variables affecting the feed pellet quality were also identified. In the single pelleting unit, the bran-CDS ratio and the pelleting temperatures were the process variables. The moisture content of the bran and CDS, the retention time during pelleting, steam conditioning, and the bran-CDS ratio were the process variables which affected the final product quality in the pilot-scale pellet mill.

## **6.2 Conclusions**

The following conclusions are drawn based on the experiments and analysis conducted in this research.

### **6.2.1 Optimizing the debranning of wheat using two laboratory equipment, the Satake and TADD mill**

1. The optimal conditions of debranning in the Satake mill, based on debranning efficiency, were 30 and 200 g sample size, 1412 and 1515 rpm rotational speed, all grit sizes, and 60 s retention time.
2. The optimal condition of debranning in the TADD mill were 200 g sample size, 900 rpm rotational speed, 50 and 80 mm grit size, and 180 and 240 s retention time. With these conditions, the TADD mill provided results almost comparable with Satake results.
3. The Satake mill provided better debranning results as compared to TADD, with high productivity, debranning efficiency, and low production time.

4. Based on the starch separation efficiency, the Satake mill provided very good results with the 200 g sample size, 1515 rpm rotation speed, 30 grit size, and 60 s retention time.

## **6.2.2 Pelleting characteristics of bran and CDS as animal feed**

### **6.2.2.1. Single pelleting of bran-CDS mixture**

1. The 90:10 bran-CDS ratio produced feed pellets of high durability and pellet density, although the physical quality of the pellets was not significantly different from the other bran-CDS ratio pellets.
2. The 90°C pelleting temperature produced feed pellets of high durability and pellet density, although the physical quality of the pellets was not significantly different from the other pelleting temperatures.
3. The moisture content of the bran-CDS mixtures reduced on pelleting and after cooling (Day 14).

### **6.2.2.2. Pilot-scale pelleting of bran-CDS mixture**

1. The moisture content of the bran-CDS mixtures reduced on pelleting and after cooling (Day 14).
2. Pelleting enhanced the bulk density of the bran-CDS pellets, and the values obtained were negatively correlated to the moisture content of the pellets. The highest bulk density was observed for 90:10 bran-CDS proportion, having lowest moisture content of  $9.81 \pm 0.13\%$

3. The three bran-CDS ratios formed highly durable pellets (durability values > 90%). The highest durability was seen in 70:30 ratio pellets ( $97.4 \pm 0.3\%$ ), although the durability values of the 70:30 and 80:20 bran-CDS ratio pellets were not significantly different.
4. Durability of the pellets was negatively correlated to pellet mill throughput, and was positively correlated to specific energy consumption.
5. The 70:30 bran-CDS mixture produced pellets with high nutrient content (18.42% crude protein, 4.38% fat, 63.80% fiber, 9.05% starch, and 18.93 MJ/kg gross energy), and physical properties.

In conclusion, this study was able to achieve the research objectives. The two debranning equipment, the Satake mill and the TADD, were used to optimize the debranning process. The debranning efficiency and the starch separation efficiency of the Satake mill showed higher potential for usage in the industrial levels. The bran from the debranning experiments and the CDS were combined, and processed to produce feed pellets. The 70:30 bran-CDS ratio produced highly durable and dimensionally stable feed pellets.



## **CHAPTER SEVEN**

### **PROJECT RECOMMENDATIONS**

The following are the recommendations for further research:

1. A detailed study of debranning using sample sizes greater than 200 g is recommended. Effect of changes in the sample sizes on the debranning efficiency and the starch separation efficiency should be studied. This will help in better utilization of the laboratory-scale results in the industrial levels.
2. In the single pelleting studies, only two parameters, i.e. the ingredient formulation and pelleting temperatures, were varied to optimize the pelleting process. Changes in the physical properties of the feed pellets, affected by parameters such as particle size and compressive force of the pelleting unit should be studied.
3. Pilot-scale pelleting was done with uncontrolled steam conditioning. The process variables (steam conditioning and retention time) in the pilot-scale pelleting should be varied to have a better understanding of their effect on the feed pellets.
4. The results from the optimization studies can be used for development of debranning and pelleting processes in the industrial levels. This would be useful in providing information for future studies. Additionally, a techno-economic evaluation can be good way to determine the economic suitability of adding the debranning and pelleting processes in ethanol production.

5. The primary focus of this study was the development of feed pellets having good nutritional and physical properties. For future works, animal studies of the bran-CDS pellets are suggested to attain conclusive results based on the practical usage of the feed pellets manufactured.

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## APPENDIX A

Table A.1 Experimental plan (Optimizing the debranning of wheat)

Materials	Wheat grains
	Satake mill
Debranning equipment	Tangential abrasive dehulling device (TADD)
Sample size (A), g	A1 = 30 A2 = 200
Rotation speed (S) for Satake mill, rpm	S1 = 1215 S2 = 1412 S3 = 1515
Grit size (G) for Satake mill, mm	G1 = 30 G2 = 36 G3 = 40
Retention time (T) for Satake mill, s	T1 = 30 T2 = 60 T3 = 90
Rotation speed (s) for TADD, rpm	s1 = 900 g1 = 30
Grit size (g) for TADD, mm	g2 = 36 g3 = 50 g4 = 80
Retention time (t) for TADD, s	t1 = 30 t2 = 60 t3 = 90

Table A.2 Statistical analysis for debranning fraction for Satake mill (30 g)

Variable	Mean sq.	F-value	Pr (>F)
Speed	1104.38	3385.3061	$< 2.2 \times 10^{-16}$ ***
Grit	19.41	59.5002	$9.418 \times 10^{-11}$ ***
Time	2358.45	7229.4408	$< 2.2 \times 10^{-16}$ ***
Replicates	3.98	12.1998	0.000865***
Speed x grit	11.17	34.2351	$1.743 \times 10^{-07}$ ***
Speed x time	191.80	587.9251	$< 2.2 \times 10^{-16}$ ***
Grit x time	1.22	3.7510	0.057124 .
Speed x replicates	0.00	0.0099	0.921085
Grit x replicates	0.37	1.1487	0.287788
Time x replicates	0.86	2.6512	0.108309
Speed x grit x time	1.12	3.4383	0.068239 .
Speed x grit x replicates	0.01	0.0303	0.862411
Speed x time x replicates	0.06	0.1698	0.681662
Grit x time x replicates	0.33	1.0109	0.318419
Speed x grit x time x replicates	0.00	0.0049	0.944575
Residuals	0.33		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.3 Statistical analysis for debranning fraction for Satake mill (200 g)

Variable	Mean sq.	F-value	Pr (>F)
Time	7355.4	1873.1804	< 2.2×10 <sup>-16</sup> ***
Rotational speed	40.9	10.4126	0.0026 **
Rotational speed x grit x time	24.7	6.2993	0.0164 *
Grit	16.8	4.2709	0.045634 *
Replicates	1.1	0.2857	0.596119
Speed x grit	8.7	2.2060	0.145725
Speed x time	12.6	3.2159	0.080889 .
Grit x time	0.0	0.0103	0.919829
Speed x replicates	0.2	0.0425	0.837829
Grit x replicates	0.9	0.2248	0.638119
Time x replicates	0.1	0.0144	0.905032
Speed x grit x replicates	0.0	0.0001	0.994123
Speed x time x replicates	0.8	0.2136	0.646628
Grit x time x replicates	1.1	0.2831	0.597798
Speed x grit x time x replicates	0.8	0.1941	0.662001
Replicates	3.9		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 ' ' significant at P= 1



Table A.4 Statistical analysis for debranning fraction for TADD mill (30 g)

Variable	Mean sq.	F-value	Pr (>F)
Grit	21.796	173.6212	$3.883 \times 10^{-16}$ ***
Time	97.295	775.0257	$< 2.2 \times 10^{-16}$ ***
Replicates	0.070	0.5601	0.4586
Grit x time	0.096	0.7622	0.3879
Grit x replicates	0.014	0.1097	0.7422
Time x replicates	0.050	0.3959	0.5328
Grit x time x replicates	0.070	0.5539	0.4611
Residuals	0.126		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.5 Statistical analysis for debranning fraction for TADD mill (200 g)

Variable	Mean sq.	F-value	Pr (>F)
Grit	741.64	113.6157	$1.393 \times 10^{-10}$ ***
Time	230.45	35.3037	$3.933 \times 10^{-06}$ ***
Replicates	0.00	0.0004	0.98470
Grit x time	33.39	5.1150	0.03306 *
Grit x replicates	0.20	0.0303	0.86318
Time x replicates	0.16	0.0245	0.87689
Grit x time x replicates	0.01	0.0018	0.96691
Residuals	6.53		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.6 Statistical analysis for starch content for Satake mill (30 g)

Variable	Mean sq.	F-value	Pr (>F)
Speed	57.709	11.7447	0.0140218 *
Grit	220.003	44.7738	0.0005403 ***
Time	64.496	13.1259	0.0110578 *
Replicate	52.326	10.6492	0.0171774 *
Speed x Grit	43.108	8.7730	0.0252197 *
Grit x time	21.580	4.3919	0.0809587 .
Speed x Replicate	0.023	0.0046	0.9482479
Grit x Replicate	3.866	0.7867	0.4092440
Time x Replicate	11.486	2.3375	0.1771590
Speed x grit x replicate	0.453	0.0922	0.7716078
Grit x time x replicate	18.780	3.8220	0.0983758 .
Residuals	4.914		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.7 Statistical analysis for starch separation efficiency for Satake mill (30 g)

Variable	Mean sq.	F-value	Pr (>F)
Speed	184.704	22.9147	0.01734 *
Grit	156.472	19.4123	0.02168 *
Time	23.207	2.8791	0.18831
Speed x Grit	121.516	15.0755	0.03027 *
Grit x time	13.326	1.6532	0.28878
Residuals	8.060		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.8 Statistical analysis for starch content for Satake mill (200 g)

Variable	Mean sq.	F-value	Pr (>F)
Speed	1102.7	41.6837	1.350 x10 <sup>-07</sup> ***
Grit	134.0	5.0638	0.03030 *
Time	3766.1	142.3685	2.026 x 10 <sup>-14</sup> ***
Replicate	0.8	0.0292	0.86519
Speed x Grit	27.2	1.0293	0.31675
Speed x time	141.2	5.3359	0.02642 *
Grit x time	89.5	3.3845	0.07363 .
Speed x Replicate	0.3	0.0131	0.90936
Grit x Replicate	0.2	0.0068	0.93462
Time x Replicate	0.8	0.0287	0.86632
Speed x grit x time	29.3	1.1089	0.29897
Speed x Grit x Replicate	2.4	0.0897	0.76623
Speed x time x Replicate	0.1	0.0026	0.95990
Grit x time x Replicate	2.0	0.0761	0.78410
Speed x grit x time x Replicate	5.2	0.1972	0.65952
Residuals	26.5		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

Table A.9 Statistical analysis for starch separation efficiency for Satake mill (200 g)

Variable	Mean sq.	F-value	Pr (>F)
Speed	904.87	18.9927	9.628 x10 <sup>-05</sup> ***
Grit	58.80	1.2341	0.2735875
Time	800.32	16.7983	0.0002105 ***
Replicate	1.48	0.0310	0.8611837
Speed x Grit	75.62	1.5872	0.2154065
Speed x time	8.89	0.1866	0.6682332
Grit x time	89.09	1.8700	0.1795111
Speed x Replicate	0.50	0.0105	0.9189444
Grit x Replicate	0.06	0.0012	0.9728039
Time x Replicate	1.50	0.0316	0.8598900
Speed x grit x time	120.49	2.5291	0.1200535
Speed x Grit x Replicate	2.08	0.0436	0.8357237
Speed x time x Replicate	0.10	0.0021	0.9639345
Grit x time x Replicate	2.92	0.0612	0.8059008
Speed x grit x time x Replicate	5.67	0.1190	0.7320173
Residuals	47.64		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 ' ' significant at P= 1

Table A.10 Statistical analysis for starch content and starch separation efficiency for TADD mill  
(200 g)

	Variable	Mean sq.	F-value	Pr (>F)
Starch content	Grit	0.504	0.0604	0.8466
	Time	104.546	12.5139	0.1754
	Residuals	8.354		
Starch separation efficiency	Grit	0.371	0.0310	0.8891
	Time	84.760	7.0722	0.2290
	Residuals	11.985		

Signif. Codes: 0 '\*\*\*' significant at P= 0.001, '\*\*' significant at P= 0.01, '\*' significant at P= 0.05, '.' significant at P= 0.1 '' significant at P= 1

## APPENDIX B

Table B.1: Statistical analysis for moisture content of pellets (single pelleting, Day 1)

Variable	Mean sq.	F-value	Pr (>F)
Temp	0.03154	0.0978	0.7671
Proportion	0.31190	0.9670	0.3706
Proportion x temp	0.04040	0.1253	0.7378
Residuals	0.32256		

Signif. Codes: 0 '\*\*\*' signif. at P= 0.001, '\*\*'signif. at P= 0.01, '\*' signif. at P= 0.05, '.' Signif. at P= 0.1 ' ' signif. at P= 1

Table B.2: Statistical analysis for changes in pellet density (single pelleting, Day 1 and 14)

Variable	Mean sq.	F-value	Pr (>F)
Ratio	97.081	25.8035	$2.288 \times 10^{-06}$ ***
Temp	26.735	7.1059	0.009232 **
Ratio x temp	10.395	2.7630	0.100237
Residuals	3.762		

Signif. Codes: 0 '\*\*\*' signif. at P= 0.001, '\*\*'signif. at P= 0.01, '\*' signif. at P= 0.05, '.' signif. at P= 0.1 ' ' signif. at P= 1

Table B.3: Statistical analysis for durability (single pelleting, Day 14)

Variable	Mean sq.	F-value	Pr (>F)
Ratio	119.286	17.8218	$5.996 \times 10^{-05}$ ***
Temp	0.253	0.0379	0.8462
Ratio x temp	0.361	0.0539	0.8169
Residuals	6.693		

Signif. Codes: 0 '\*\*\*' signif. at P= 0.001, '\*\*'signif. at P= 0.01, '\*' signif. at P= 0.05, '.' Signif. at P= 0.1 ' ' signif. at P= 1

Table B.4: Statistical analysis of bulk density of bran-CDS pellets (pilot-scale pelleting, Day 1)

<b>Variable</b>	<b>Mean sq.</b>	<b>F-value</b>	<b>Pr (&gt;F)</b>
Ratio	299.91	$2.749 \times 10^{29}$	$<2 \times 10^{-16}$ ***
MC	53.49	$4.903 \times 10^{28}$	$<2 \times 10^{-16}$ ***
Retention_time	13.55	$1.242 \times 10^{28}$	$<2 \times 10^{-16}$ ***
Ratio x MC	0.00	$1.437 \times 10^{01}$	0.0322 *
MC X Retention_time	0.00	$4.141 \times 10^{00}$	0.1347

Signif. Codes: 0 '\*\*\*' signif. at P= 0.001, '\*\*'signif. at P= 0.01, '\*' signif. at P= 0.05, '.' Signif. at P= 0.1 ' ' signif. at P= 1

Table B.5: Statistical analysis of durability of bran-CDS pellets (pilot-scale pelleting, Day 1)

<b>Variable</b>	<b>Mean sq.</b>	<b>F-value</b>	<b>Pr (&gt;F)</b>
Ratio	17.716	30.156	0.0119 *
MC	1.672	2.845	0.1902
Retention_time	0.327	0.556	0.5100
Ratio x MC	0.599	1.020	0.3870
MC X Retention_time	0.146	0.248	0.6525

Signif. Codes: 0 '\*\*\*' signif. at P= 0.001, '\*\*'signif. at P= 0.01, '\*' signif. at P= 0.05, '.' signif. at P= 0.1 ' ' signif. at P= 1